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NWC Standard Methods for Determining Thermal Properties of Propellants and Explosives

by
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MARCH 1980

NAVAL WEAPONS CENTER
CHINA LAKE, CALIFORNIA 93555

Naval Weapons Center

AN ACTIVITY OF THE NAVAL MATERIAL COMMAND

FOREWORD

This report describes a series of tests that have been developed at the Naval Weapons Center (NWC), China Lake, California, to define thermal properties of a material. These tests document one phase of a continuing research program at NWC in support of the determination of thermal properties of propellants and explosives. The work described has been funded under Naval Sea Systems Command Work Assignment N0002479WR9B902.

This report has been prepared primarily for timely presentation of information and is released at the working level.

This report has been reviewed for technical accuracy by Dr. Gregory Vernon.

Approved by
C. L. SCHANIEL, *Head*
Ordnance Systems Department
1 July 1979

Under authority of
W. B. HAFF
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(U) As a part of the qualification of explosives and propellants for Navy use, the thermal properties of explosives and propellants are required to be able to make a prediction of the safety of these materials in production, transportation, storage, and use. A number of methods for determining the thermal properties of materials have been developed and standardized over the years. These fall into three groups: (1) laboratory tests using less than 1 gram of material: DTA/TGA, DSC, and specific heat and isothermal composition tests; (2) intermediate size tests using 1 to 5 pounds of material: thermal diffusivity, and fast and slow cook-off tests, and (3) full-scale fast cook-off. Examples are used to illustrate the data available from each test.

CONTENTS

INTRODUCTION

Explosives and propellants used in Navy munitions are required to be qualified for performance and for safety in their manufacture, handling, storage, and use. Tests to be performed to qualify an explosive or propellant are described in OD-44811[1] and MIL-STD-1648(AS).[2] Among the tests for safety of an explosive or propellant are those for thermal stability, self-heating, and cook-off. A series of tests to define the thermal properties of a material have been developed at the Naval Weapons Center (NWC) and are being performed on a regular basis. These fall into three groups:

1. Laboratory scale tests using less than 1 gram of material to define thermal properties of that material, including thermal stability and incompatibilities.
2. Intermediate scale tests using 1 to 5 pounds of an explosive or propellant. These include:
   a. Test for thermal diffusivity.
   b. A slow cook-off (SCO) series to define the self-heating and storability properties.
   c. Fast cook-off test, small-scale cook-off bomb (SCB) to define the response of the material to a rapid temperature rise, such as occurs in a fire.
3. Full-scale munitions tests to determine the response to rapid heating in a fuel fire.

This report describes these standardized procedures.

LABORATORY TESTS FOR THERMAL STABILITY

A series of tests is conducted to determine thermal properties of a material and to supply the data necessary to estimate a characteristic "critical temperature" for heat balance and to estimate a time-to-reaction for the various configurations and thermal conditions in storage and in use. The required properties include density, specific heat, thermal conductivity, thermal diffusivity, heat released in the reactions leading to cook-off, and the chemical reaction rate parameters, activation energy, and

[1] NAVORD OD 44811, Vol. 1, "Safety and Performance Tests for Qualification of Explosives," 1 January 1972.

[2] NASC MIL-STD-1648(AS), "Criteria and Test Procedures for Ordnance Exposed to an Aircraft Fuel Fire," 28 March 1974.

frequency factor. Thermal data needed to define these properties are obtained in the form of differential thermal analyses (DTA), thermogravimetric analyses (TGA), differential scanning calorimetry (DSC), thermal diffusivity, and thermal stability measurements.

DIFFERENTIAL THERMAL AND THERMOGRAVIMETRIC ANALYSES

Simultaneous DTA and TGA are obtained using a Mettler Instrument Co. Thermoanalyzer-2 (T/A-2).* This instrument measures the temperature of a sample, relative to a known reference material. The sample and the reference material are placed in crucibles mounted in an oven whose temperature is changed at a linear rate. Reactions, phase transitions, and decompositions produce deflections in the DTA record that are proportional to the heat involved. In the T/A-2 (Figure 1), the crucibles are mounted on an electro-microbalance so that a simultaneous weight of the sample present is obtained. An instantaneous rate of weight loss, the slope of the tangent to the TGA curve, derivative thermogravimetry (DTG), is also recorded as a function of oven temperature. Temperatures of the oven and sample are measured with platinum–10% rhodium in platinum thermocouples. About 10 milligrams of indium metal is placed in the reference crucible to provide a calibration point for the temperature record at the melting point of indium (156°C). Scales supplied by the T/A-2 manufacturer are used to locate points on the temperature record.

Procedure

Procedures used in setting up an instrument run are described in the manufacturer's instruction book. Sample size, heating rate, temperature scale, and recorder ranges are chosen variables. A typical set of ranges are:

Sample weight	10-50 mg
TGA range	100 mg, full scale
DTG range	5 mg/min, full scale
Atmosphere	Air
DTA range	100 μV, full scale
Reference material	Indium
Weight	7-10 mg
Heating rate	3°C/min
Thermocouple	Pt/Pt-Rh10
Temperature	2 Mv full scale
Recorder chart speed	12 in/hr

Examples of reduced records are shown in Figures 2 and 3.

* Mention of a specific manufacturer's instrument does not constitute an endorsement or recommendation of that instrument by the U.S. Government.

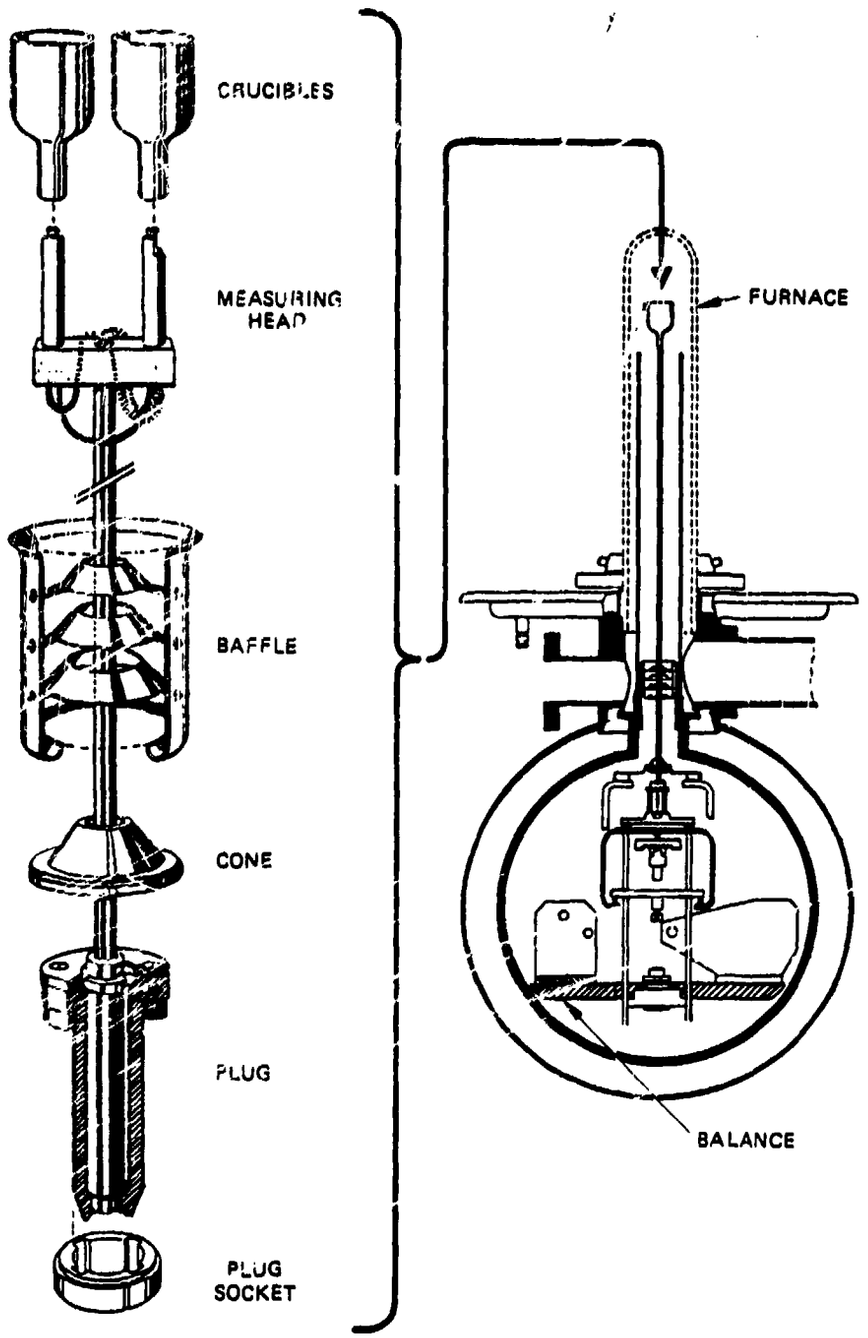

FIGURE 1. Mettler Instrument Co. Thermoanalyzer-2.

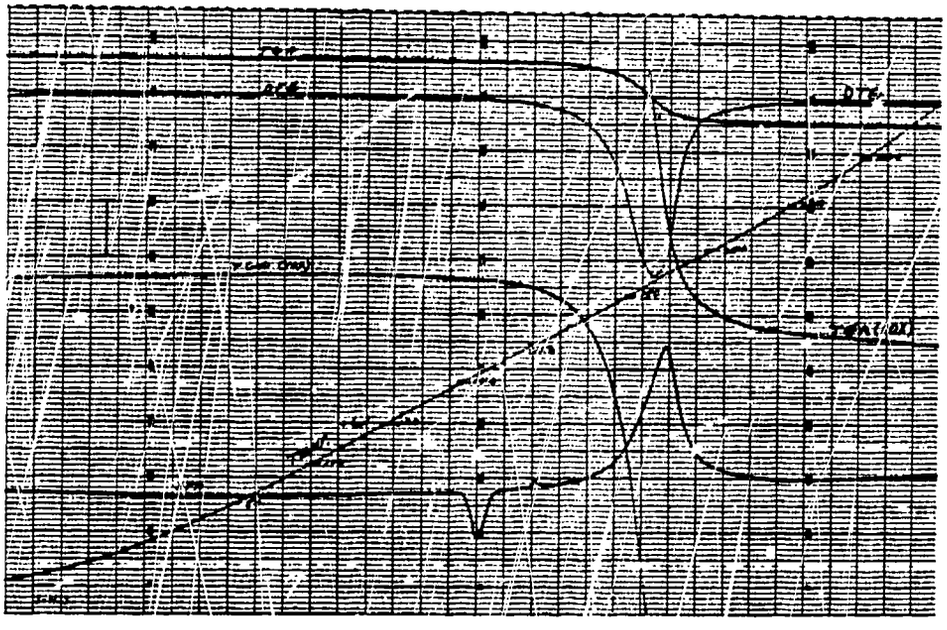

FIGURE 3. Thermal Patterns of PETN (Reference Sample for H. Stanton, 1-8-73) at Heating Rate of 3°C/min. (Sample wt.: 31.75 mg; run no. 10-11-3)

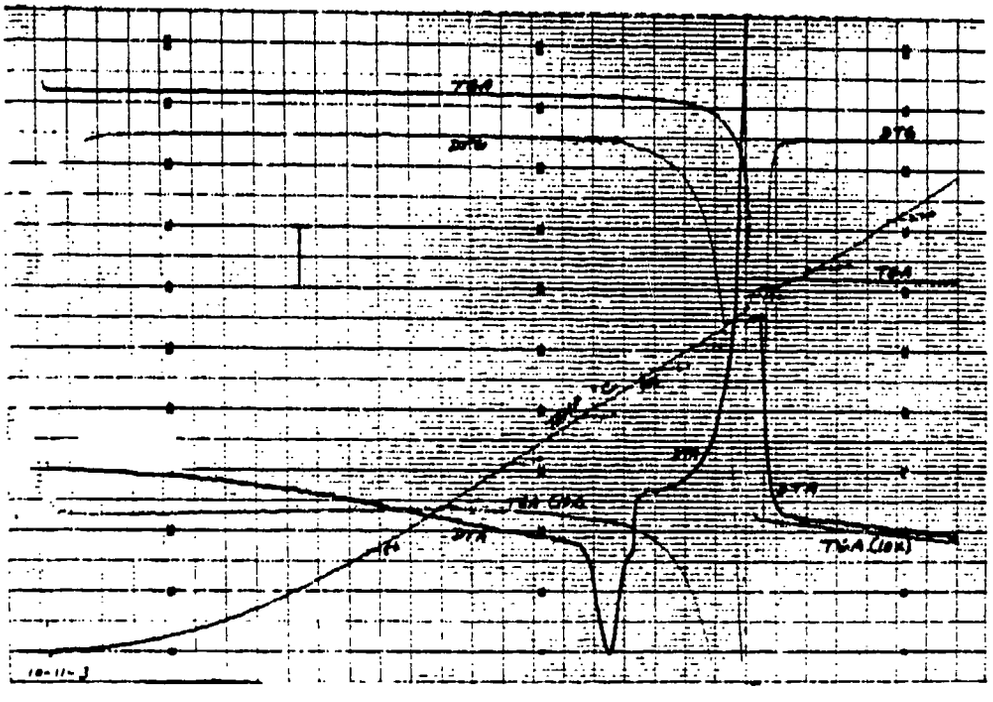

FIGURE 4. Thermal Patterns on PETN at Heating Rate of 3°C/min. (Sample wt.: 11.9 mg; run no. 2-74-3)

Evaluation

The oven temperature trace of the T/A-2 record is calibrated by the small exotherm produced by the melting indium in the reference crucible; this point is 156°C. Using the scales provided by the T/A-2 manufacturer, tick marks are made on the temperature trace to aid in reading the chart. In an evaluation, such as from the reduced records of Figure 2, the following features of the pattern would be listed for the sample PETN:

Sample	PETN
Sample weight	11.9 mg
First weight loss (0.03 mg)	127°C
Melting point, PETN	141°C
Exotherm initiate	155°C
Exotherm peak	190°C
Total weight loss	11 mg

The first weight loss at 127°C, as read on the TGA (x10) trace, suggests that PETN has an appreciable sublimation pressure or that the decomposition reactions are occurring at an observable rate in the solid state at these temperatures. Many explosive and propellant patterns show a "burst" reaction in which a sizable fraction of the sample disappears suddenly. For example, PETN does show a burst reaction when a larger sample (20-40 milligrams) is used (Figure 3). The temperature at which the burst occurs is a function of the sample size and the heating rate; for PETN, a 31.77 milligram sample showed a burst at 186°C. The burst reaction temperature could be considered an autoignition temperature, *but only* for that size of sample, in that configuration, and at that heating rate.

Thermal patterns for mixtures will have information on the compatibility of the components of the mixture. An incompatibility is indicated by a shift in reaction temperatures to some lower temperature. An increase in reaction rate or lowering of the activation energy is also indicative of an incompatibility.

Data Reduction

There are numerous methods of extracting chemical reaction rate data from DTA and TGA traces.[3-7] All of the methods have the object of deriving the chemical

[3] M. J. Balarin, *J. Thermal Analysis*, Vol. 12 (1977), p. 169.
[4] H. R. Petty, R. T. Arakawa, and J. K. Baird, *J. Thermal Analysis*, Vol. 11 (1977), p. 417.
[5] A. R. Zatsepin, A. A. Fetieu, and I. A. Dimitriev, *Russian Journal of Inorganic Chemistry*, Vol. 18 (1973), p. 1533.
[6] A. N. Coats and J. P. Redfern, *Nature*, Vol. 201 (1964), p. 68.
[7] "Thermal Analysis," edited by R. B. Schwenker and P. D. Garn, *Academic Press*, Vol. 2, New York, 1969.

reaction rate parameters—activation energy, E^*; and frequency factor, A,—of the Arrhenius equation:

$$k = A \exp(-E^*/RT) \tag{1}$$

where

k = specific reaction rate constant
T = temperature, K
R = universal gas constant, 1.987 cal/mol·K

The specific reaction rate constant is the proportionality constant in the mass action law relating reaction rate to concentration of reactants:

$$-\frac{dc}{dt} = kc^n \tag{2}$$

n is called the order of the reaction and is the observed dependence of the rate of disappearance of reactants, $-dc/dt$, on the concentration of the reactants. In terms of weight of reactants:

$$-\frac{1}{w_0} \cdot \frac{dw}{dt} = k\left(\frac{w}{w_0}\right)^n \tag{3}$$

Since the reactions of concern here are either zero order or the extent of reaction is small, Equations 1 and 3 can be combined to give:

$$-\frac{dw}{dt} = Aw_0 \exp(-E^*/RT) \tag{4}$$

which is directly applicable to the DTG trace on the T/A-2 record. A plot of the logarithm of the DTG deflections, as milligrams per second per milligram of sample versus the reciprocal of the absolute temperature at that point, should produce a straight line whose slope is $-E^*/R$. Figure 4 is such a plot of data derived from T/A-2 traces in Figures 2 and 3. From Figure 4, the activation energy is 50.3 kcal/mol and the frequency factor is 2.1×10^{20} s^{-1} for a 11.90 milligram sample. The larger 31.75 milligram sample of Figure 3 produced an activation energy of 44.0 kcal/mol and a frequency factor of 1.05×10^{18} s^{-1}. The low temperature reactions have activation energies of 29.1 kcal/mol for a 11.9 milligram sample and 21.2 kcal/mol for a 31.74 milligram sample.

DIFFERENTIAL SCANNING CALORIMETRY

A Differential Scanning Calorimeter (DSC), Perkin-Elmer Model 1B, is used for obtaining thermal scan rate, specific heat, and heat of reaction data. The instrument measures the difference in the amount of energy required to maintain a

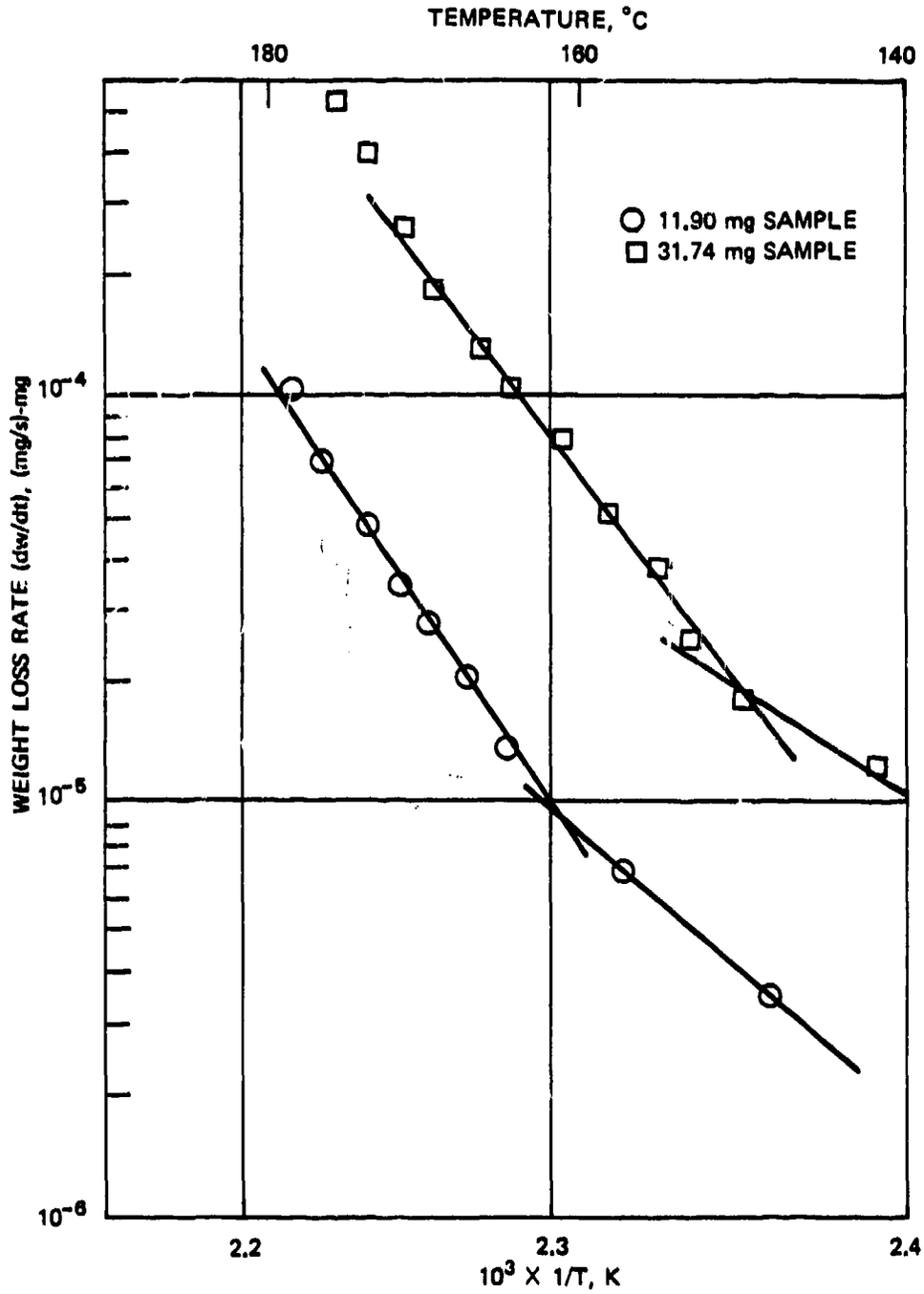

FIGURE 4. DTA/TGA Weight Loss Rate Data, PETN.

given heating rate in a sample and in an inert reference. Since this is a true energy measurement, the area under a deflection in the recorder trace is proportional to the heat of reaction and can be calibrated by a known reaction. The Kissinger method, the change in the temperature of maximum reaction with thermal scan rate, provides a means for obtaining reaction kinetic parameters. Many explosives and propellant react suddenly in the T/A-2, leaving the DSC as the only means of obtaining the kinetic parameters of a "burst" reaction.

Procedures

Procedures for the operation of the DSC are provided in the manufacturer's instruction manual. With samples of 1 to 2 milligrams, thermal scan rates of up to 40°C/min can be obtained. The usual thermal scan rate series is 10°C/min, 20°/min, and 40°C/min. Figure 5 shows the recorder traces for a DSC scan rate series with PETN.

In the lower temperature regions, the reactions in these materials are very slow so that the material appears to be essentially inert. Thus, the energy input necessary to maintain a heating rate is proportional to the heat capacity of the sample. Specific heat is determined by comparing the energy input for a reference material with that of the sample. (The procedure is detailed in the manufacturer's instruction manual.)

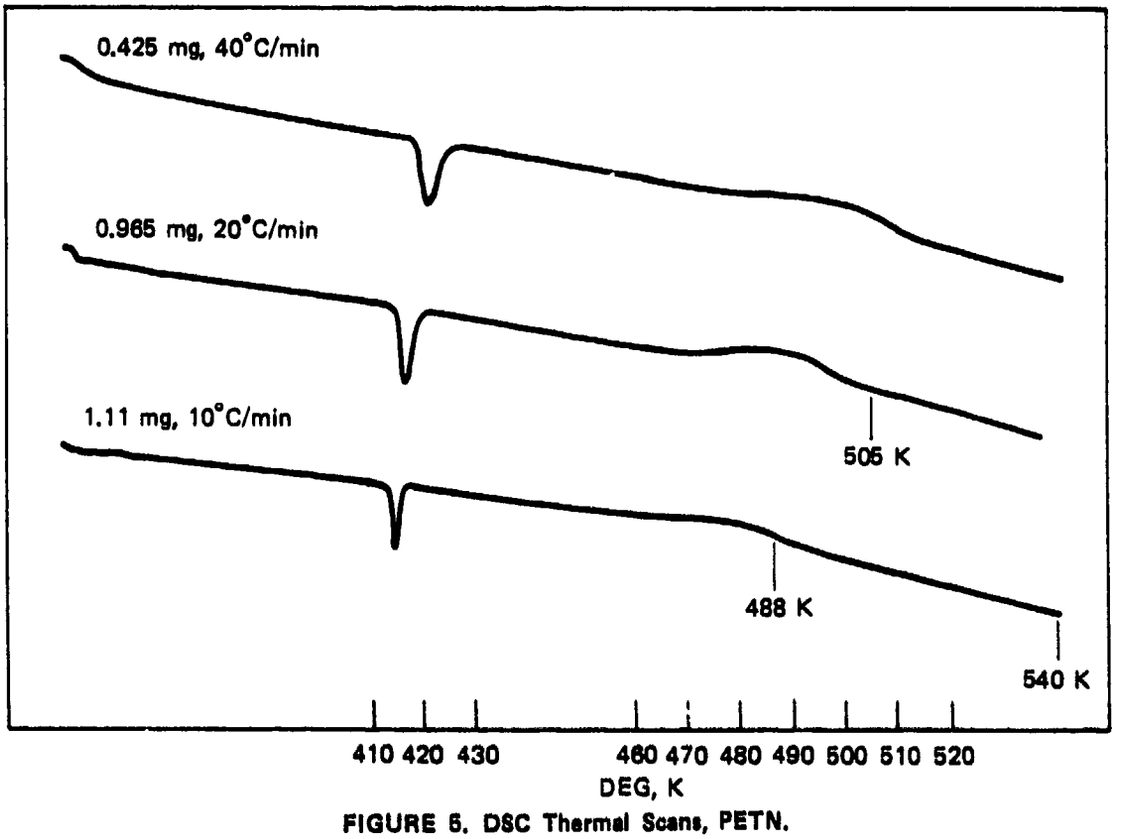

FIGURE 5. DSC Thermal Scans, PETN.

Data Reduction

Kissinger[8,9] derived an equation relating heating rate to the temperature of maximum reaction:

$$\frac{\phi}{T_m^2} \cdot \frac{E^*}{R} = A \exp(-E^*/RT_m) \tag{5}$$

where

ϕ = heating rate
T_m = temperature of maximum deflection of the DSC recorder trace, K

A plot of log $n\ \phi/T_m^2$ versus $1/T_m$ produces a line whose slope is $-E^*/R$. The frequency factor is obtained from the values of ϕ/T_m^2 and $1/T_m$ for a point on the line.

As an example of this means for determining the Arrhenius reaction rate parameters, the DSC record for a thermal scan rate series with PETN is given in Figure 5. The derived data are:

ϕ, °C/min	T_{max}, K	ϕ/T_m^2	$1/T_m$
10	478	7.3×10^{-7}	2.092×10^{-3}
20	488	1.40×10^{-6}	2.049×10^{-3}
40	501	2.7×10^{-6}	1.996×10^{-3}

which, when plotted (Figure 6), produces a line whose slope and intercept give an activation energy of 26.9 kcal/mol and a frequency factor of $2.06 \times 10^{10}\ s^{-1}$. If the DSC and T/A-2 instruments are recording the effects of the same reactions, varying the heating rate in the T/A-2 should show the same results as with the DSC. A discrepancy between DSC and DTA/TGA rate data (Figure 6) indicates that a different reaction is being measured.

Specific heat is determined by a simple ratio of deflections and specific heat-weight of sample products of a known material and the sample:

$$\frac{d_1}{d_2} = \frac{c_1 w_1}{c_2 w_2} \tag{6}$$

An example of this procedure is given in Figure 7, using PETN and a piece of sapphire as the known substance. An average value of 0.27 cal/g-°C for the specific heat of PETN was obtained from these traces.

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[8] H. E. Kissinger. *Journal of Research*, National Bureau of Standards, Vol. 57 (1956), p. 217.
[9] H. E. Kissinger. *Journal of Analytical Chemistry*, Vol. 29 (1957), p. 1703.

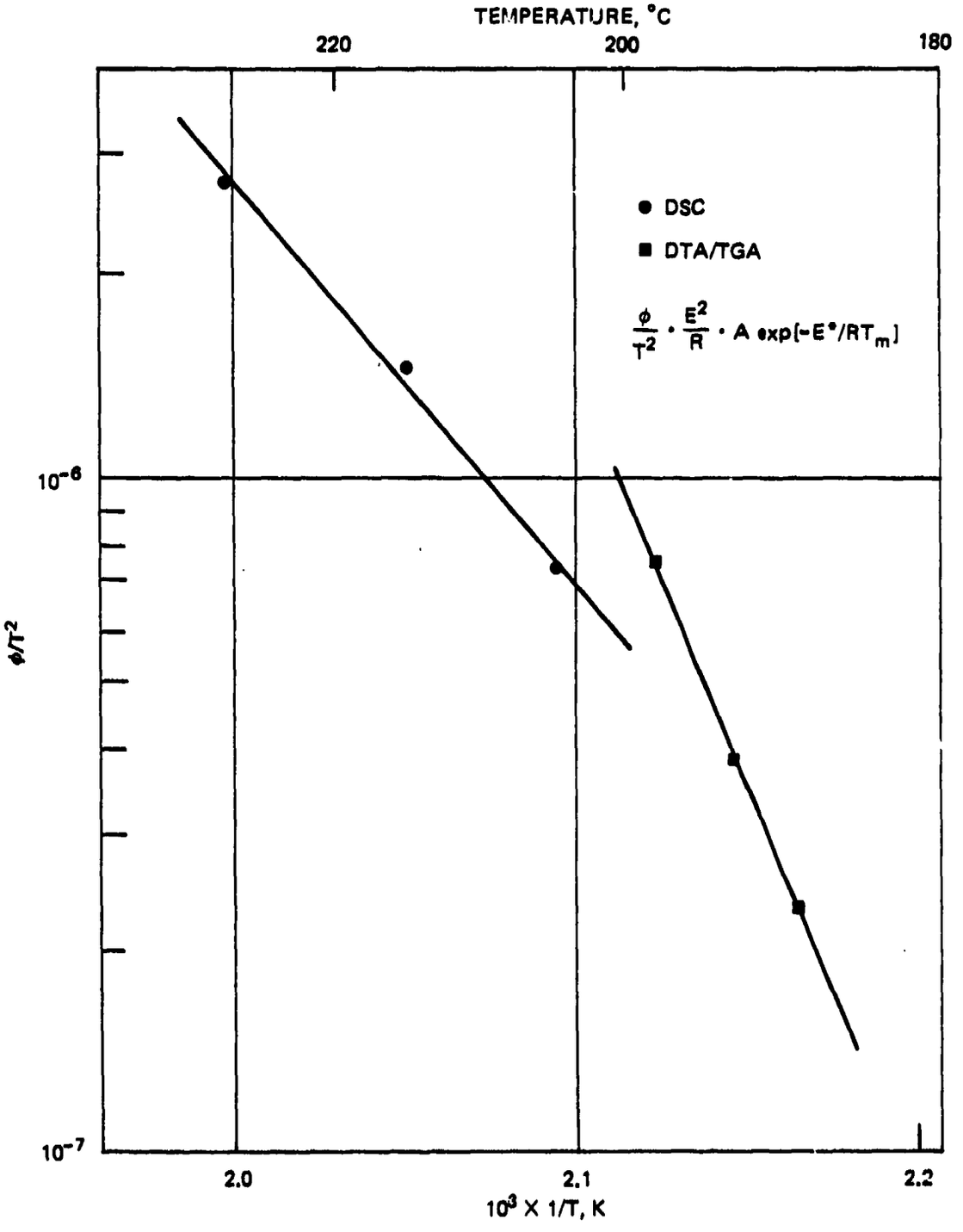

FIGURE 6. PETN Thermal Scan Rate Data.

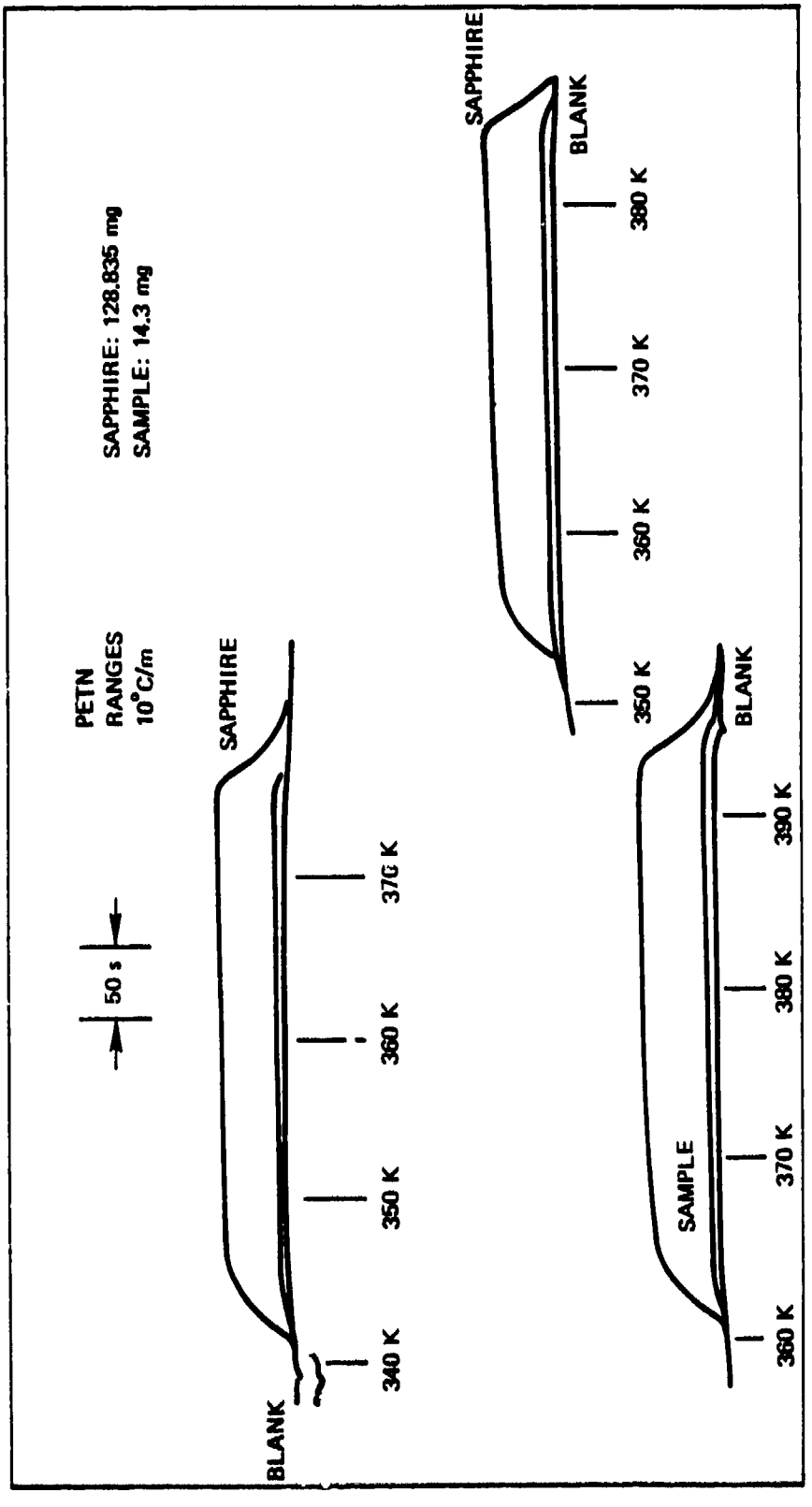

FIGURE 7. Specific Heat Determination for PETN.

THERMAL STABILITY TEST

A third laboratory procedure that produces data on the thermal properties of materials is thermal decomposition in a closed system, followed by an analysis of the gases produced. The test is conducted in a 45-millimeter pressure bomb (Parr Instrument Co.). The bomb is fitted with a valve block and pressure transducer. Aluminum block ovens with holes machined to receive the Parr bombs are used to hold the bomb at a selected temperature. Controllers (Minneapolis-Honeywell) are used to maintain the temperature within 1°C. Multipoint recorders monitor and record the time-pressure history of the sample.

Procedure

A glass vial that just slips into the 45-millimeter Parr bomb is used to contain a 1-gram sample of material. After assembling the bomb, the system is evacuated and filled with argon a number of times to flush out the air. The argon pressure is then increased to 5 to 10 atmospheres, and the valves closed for an overnight leak test. After the leak test, the pressure in the bomb is released to 1 atmosphere at ambient temperature. The bomb is then inserted into a preheated aluminum block oven and the pressure recording is started. The time-pressure recording is continued until the pressure stabilizes, at which point either the oven power is cut off or the bomb is moved to a cold oven; recording is continued until bomb temperature returns to ambient. Instrumentation for an analysis of the gases produced include a gas chromatograph, mass spectrograph, and an infrared spectrophotometer. Standard methods for the operation of those instruments are used. Finally, the bomb is disassembled and the residue weighed and inspected.

Data Reduction

The data produced in this test consist of a recorder chart of time-pressure transducer output at a particular temperature, weight loss during the test, and analysis of the residual gas in the small pressure bomb. Since the bomb at the start of the test was at 1 atmosphere of argon at ambient temperature, the initial pressure of the oven temperature can be easily found, with sufficient precision, from the ideal gas law. The increase in pressure is directly proportional to the amount of gas produced by the decomposition of the sample. The change in pressure on cooling is a measure of the amount of water produced in the reaction. Gas analysis, total gas produced, and weight loss in the sample give an indication of the particular decomposition mechanisms.

Reaction kinetic data can be obtained from this procedure by a number of techniques of which the simplest is the half-life of the reactions—the time at which one-half of the total pressure increase has occurred. For a first order reaction, the specific reaction rate constant, k_1, is:

$$k_1 = \frac{0.693}{t_{1/2}} = A \exp(-E^*/RT) \tag{7}$$

while a zero order reaction rate constant is:

$$k_0 = \frac{0.5}{t_{1/2}} = A \exp(E^*/RT) \tag{8}$$

Half-life determinations at three or more temperatures allow a plot of log n k versus $1/T$ producing a line whose slope is $-E^*/R$.

ESTIMATION OF THE "CRITICAL" TEMPERATURE FOR SELF–HEATING

Using only laboratory scale data, a first estimate can be made of the "critical" temperature for heat balance. The equation used (Appendix A) is:

$$T_{cr} = \frac{E^*}{2.303\, R \log\left[\dfrac{a^2\, QAE^*}{c\alpha R T_{cr}^2 \delta}\right]} \tag{9}$$

All of the terms in the equation have been determined except the heat of reaction, Q, and the thermal diffusivity, α. The heat of reaction used in this equation usually amounts to one-third to one-half of the heat of explosion; that is, 300 to 600 cal/g. The range of thermal diffusivity value for most of these materials is from 8×10^{-4} to 2×10^{-3} cm^2/s. Since both of these terms appear in the equation in the logarithm function, the estimate is not too seriously affected by the particular values used. Equation 9 is easily solved as a rapidly converging iteration on values of T_{cr}. OD 44811 (footnote 1) has the requirement of a critical temperature of not less than 80°C for any size of interest.

INTERMEDIATE SIZED DETERMINATIONS

The intermediate sized determinations are those that use a few pounds of an explosive or propellant. At this sample level, three procedures are commonly run: (1) a determination of the thermal diffusivity of the material, (2) a SCO series for self-heating, and (3) a fast cook-off series in a SCB, for behavior in a fire. The larger sample size in these determinations requires that precautions be taken to prevent injury or damage in the event of a violent reaction. At NWC, the firing bay for small-scale tests consists of an armor steel cylinder about 8 feet long, 5 feet in diameter and 4 inches thick, closed at one end with sheet piling and rock. The open end is closed with sandbags during any test. All explosive operations are done remotely with appropriate monitors and recorders.

THERMAL DIFFUSIVITY

A thermal diffusivity measurement allows one to obtain thermal conductivity data from a non-steady state experiment, thereby avoiding the often complex, steady state methods needed for the direct measurement of thermal conductivity. Thermal diffusivity, α, is defined as

$$\alpha = \frac{\lambda}{\rho c} \tag{10}$$

or thermal conductivity divided by the heat capacity per unit volume. As is indicated in Appendix A, the temperature difference, ΔT, between the center and the surface of a cylindrical sample of radius a at some heating rate, dT/dt, are the data required for the thermal diffusivity:

$$\alpha = \frac{a^2}{4\Delta T}\frac{dT}{dt} \tag{11}$$

Procedure

To provide the necessary conditions, a cylindrical, vertical oven is used. The oven consists of a heavy walled aluminum tube 8 to 10 inches long of any convenient inside diameter from 1-1/2 to 2-1/2 inches, welded to an aluminum base plate. A bead thermocouple is suspended at the center of the sample. A second bead thermocouple is placed in a shallow hole and peened securely into the aluminum wall. After loading, band-type heaters are fitted to the cylinder and the assembly placed in the firing bay. A controller (Minneapolis-Honeywell) is used to produce a constant heating rate in the aluminum wall. A two-pen continuous recorder monitors both of the measuring thermocouples. More than one heating cycle can be run if the sample is not taken to decomposition temperature. At the completion of the measurements, the sample is destroyed by raising the oven temperature until the sample ignites and burns out. Alternately, the temperature rise data, obtained as the sample comes up to the oven temperature in the SCO experiments, can be used to estimate a thermal diffusivity.

Data Reduction

A plot is made of the time-temperature records of the two measuring thermocouples. Equation 11 describes the measurements to be made on this plot. These are the heating rate from the slope of the curves and the temperature difference between the center and the skin at the point at which the slope was measured. As a simplification, choosing the range of the slope measurement to be the same as the temperature difference, $dt = \Delta T$, Equation 11 reduces to

$$\alpha = \frac{a^2}{4\Delta t} \tag{12}$$

where Δt is the difference in time between the two records. As an example of thermal diffusivity measurement, data for an experimental explosive is plotted in Figure 8.

SELF-HEATING–SLOW COOK-OFF

Among the qualifications of explosives for Navy use is a requirement that any size of explosive billet in use shall not self-heat more than 1°F (more than 0.5°C) at any temperature below 160°F (71°C). Alternately, the requirements specify that (1) the characteristic "critical" temperature of the explosive in the largest weapon that would use the explosive shall be greater than 80°C, or that (2) the oven temperature for cook-off in 500 days in that weapon shall be greater than 85°C. A prediction of the 500-day cook-off temperature can be made with some confidence from SCO data. In the SCO test, a time-to-reaction measurement is made of intermediate size samples at temperatures selected to produce a cook-off time in the days-to-weeks range. A good prediction can be made using three or more samples in each of three or more

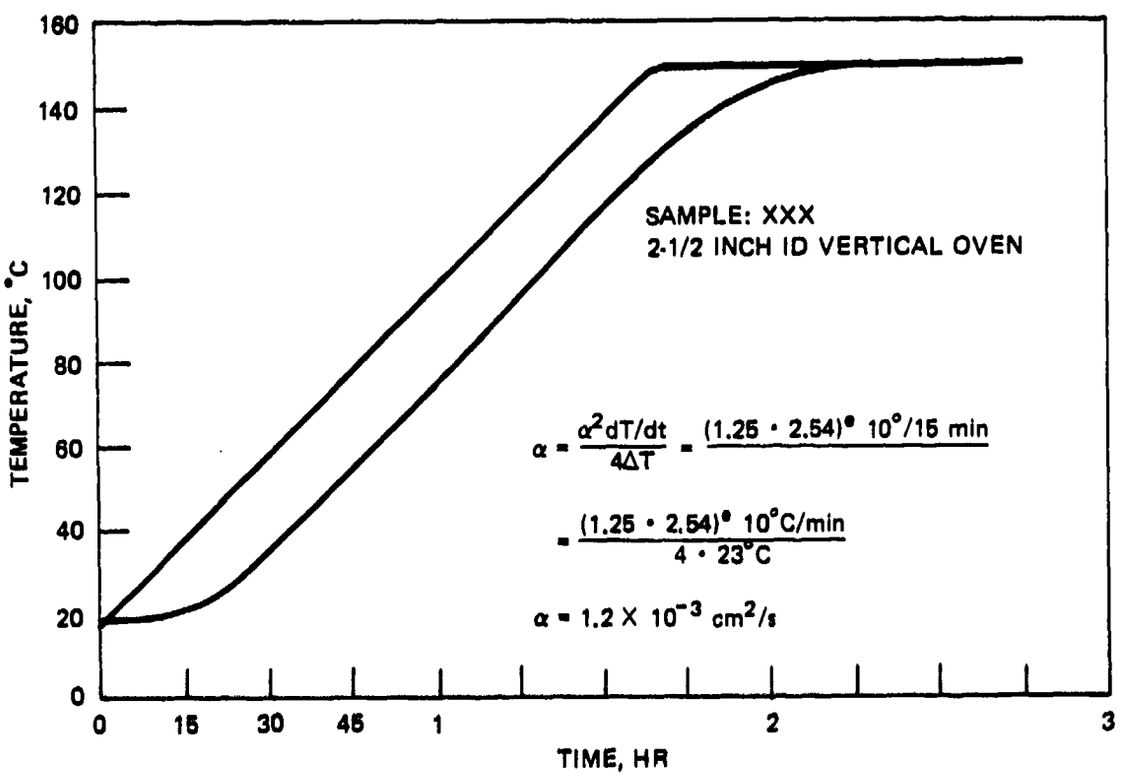

FIGURE 8. Thermal Diffusivity.

sizes to define the slopes of the lines required for the extrapolation. Alternately, and with some loss in confidence, an extrapolation can be made using data from the cook-off of three or more samples of one size.

Experimental Procedure

The ovens used for the SCO tests at NWC consist of heavy-walled aluminum or steel tubing with an 8- to 10-inch inside diameter and about 4 feet long fitted with curved strip heaters and mounted in a foil-lined 2- by 2- by 4-foot Transite box. The heaters are connected in series-parallel to obtain an electrical rating of about 4 kilowatts for the oven. Recorder-controllers (Minneapolis-Honeywell) are used to maintain oven temperature by using a chromel-alumel (Type K) thermocouple peened into the furnace wall under one of the center heaters. Small glass cloth pillows are used to close the ends of the tubing while providing a quick release for gasses produced in the cook-off reaction.

Samples for the SCO test are usually supplied in the form of cylinders but are not restricted to this form. Cast explosive or propellant samples are prepared by casting directly into aluminum-foil-lined cartons (e.g., ice-cream cartons of 3.25, 5, and 9 inches in diameter) fitted with a thermocouple at the center. Pressed samples are usually supplied as pairs of shorter cylinders that can in turn be pressed together with a thermocouple at their center. The cylindrical samples are fitted with a plate-type thermocouple on the surface of the bare explosive or propellant and tightly wrapped with two layers of 2.5-mil-thick aluminum foil. A third plate-type thermocouple is placed under the outermost layer of foil. A fourth thermocouple, usually a bead-type, is fitted to project 2 to 4 inches from the sample so as to measure the actual oven temperature. To hold the sample near the center of the oven, the sample is fastened with fiberglass tape to an inert spacer. Figure 9 shows a sample ready for the oven.

After the oven stabilizes at the selected temperature and the thermocouples have been connected to the recording system and checked out, the thermocouples are disconnected while the sample is inserted into the oven. The glass cloth pillows are put back in place and the thermocouples are reconnected. Pen-drag or multipoint recorders continuously monitor the sample and oven temperatures to supply the needed time-temperature records. The temperature selected for the initial SCO experiment is taken as the first weight loss temperature on the DTA/TGA record. Subsequent run temperatures are selected to give a spread of times to cook-off from 1 day or less to 2 to 3 weeks. In each case the sample is held at the selected temperature until the sample self-ignites and burns.

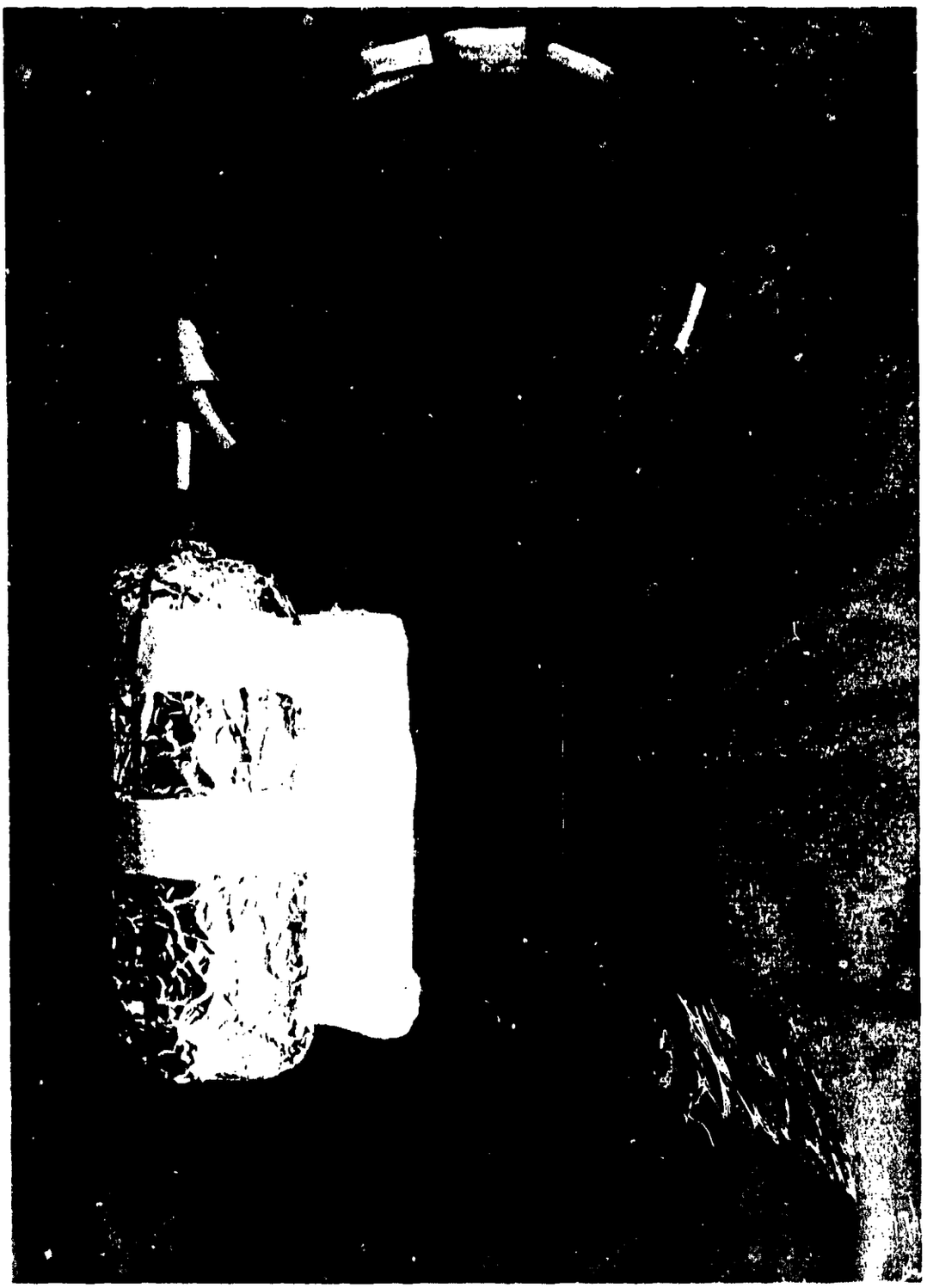

FIGURE 9. Slow Cook-Off Sample Ready for the Oven. (Neg. LHL 209094)

Data Reduction

A plot of time versus temperature is made to condense the recorder charts to manageable size. The original record is examined in two regions in particular. During warm-up a value of the thermal diffusivity, α, of the material is obtained from the time versus temperature records of the thermocouples located in the center of the sample and on the sample surface. Equation 11 suggests the measurements to be made on the record. At a sample radius, a, and some skin temperature, the temperature difference, ΔT, between the center and skin and the slope of the skin time versus temperature record, dT/dt, are measured and α calculated. The thermal conductivity, λ, of the sample follows from the definition of thermal diffusivity:

$$\lambda = \rho c \alpha \tag{13}$$

with density, ρ, and specific heat, c.

A correction to the total time in the oven is made for the amount of reaction occurring during the warm-up period to obtain an equivalent time-to-reaction at oven temperature. The correction is made by calculating a time-zero such that the same amount of reaction would occur at oven temperature that occurred during warm-up. The amount of reaction occurring during warm-up is calculated by summing the fraction reacted during the five 10-degree steps before the center of the sample reaches oven temperature. Since the fractions are small the zero order equations (1, 2) are used as:

$$f = A\ t \exp(-E^*/RT) \tag{14}$$

where T is the absolute temperature at the middle of each 10-degree segment or the oven temperature for the time-zero computation (Figure 10).

The essentially adiabatic rise of the center temperature leading to the cook-off can be used to obtain chemical reaction rate parameters. The equation obtained by Longwell[10] (Appendix A) for this situation is:

$$\rho c\ dT/dt = \rho Q k = \rho Q A \exp(-E^*/RT) \tag{15}$$

A plot of the slopes of the center time-temperature trace, dT/dt, at a temperature, T, as log dT/dt versus $1/T$ should have a slope of $-E^*/2.3R$ and an intercept of log AQ/c (Figure 11).

[10] Aerojet-General Corp. *Determination of Self-Heating Reaction Kinetics*, by P. A. Longwell, El Monte, Calif., AGC, July 1965. (AGC TM 865.)

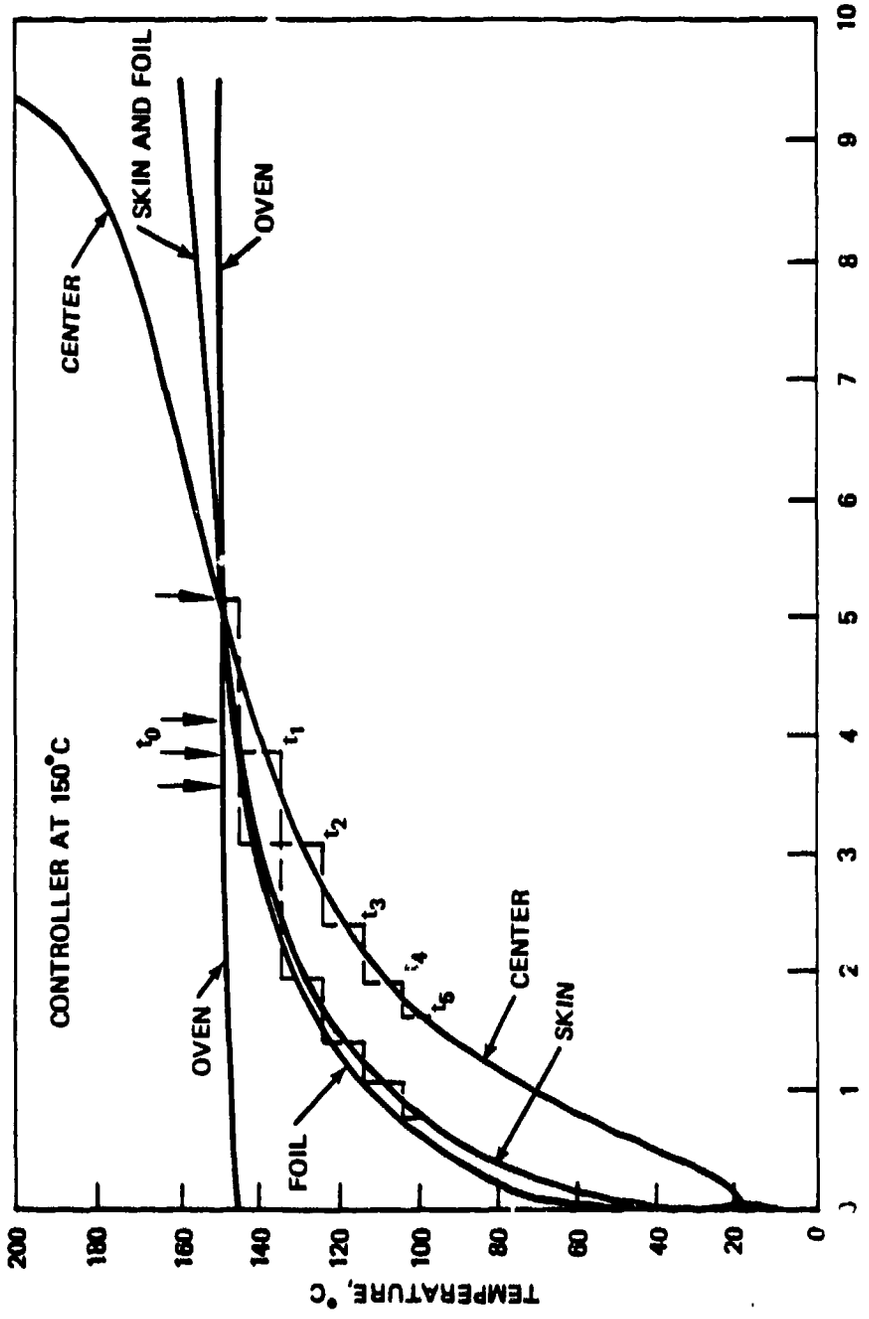

FIGURE 10. Data Controller at 150°C.

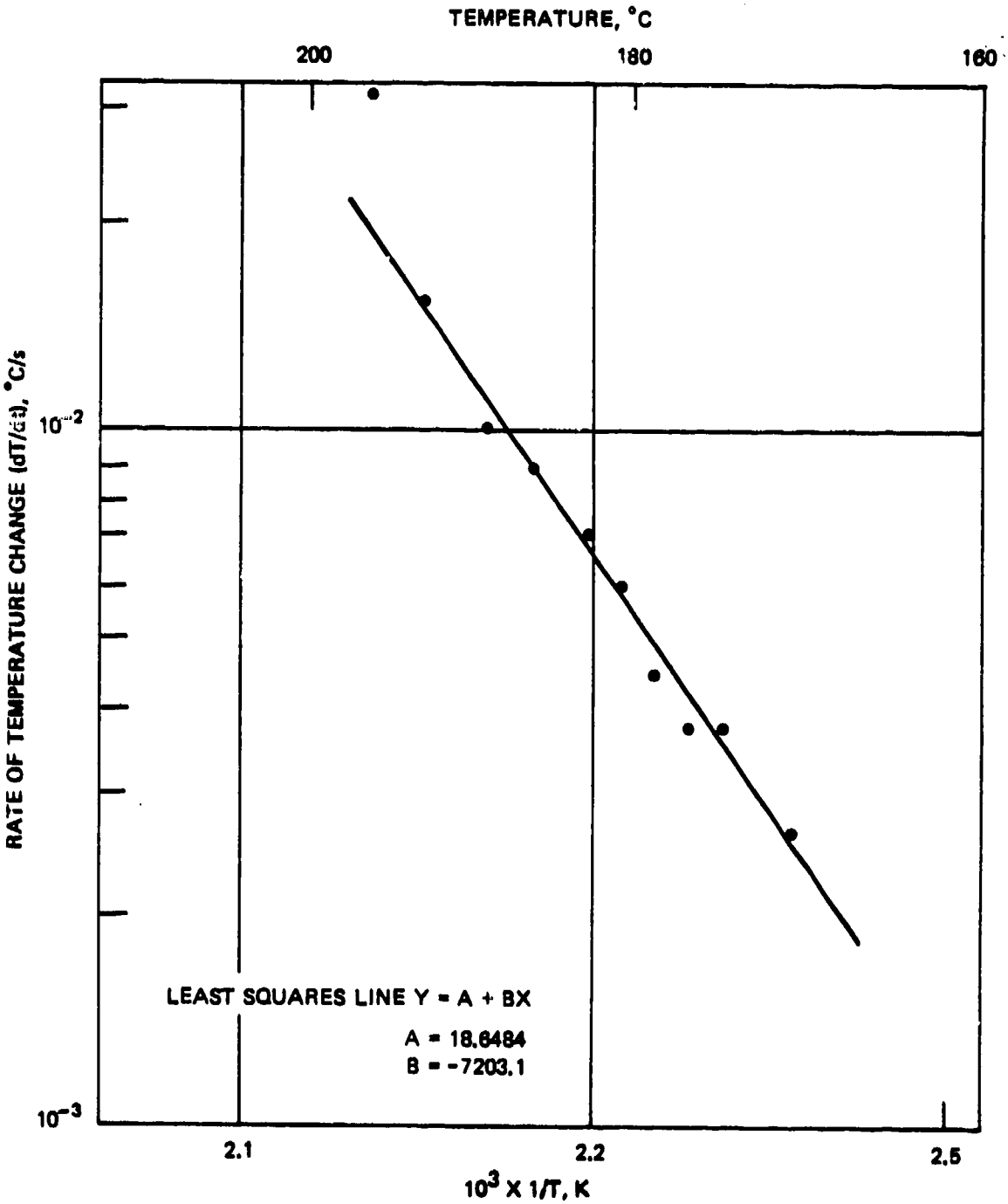

FIGURE 11. Adiabatic Temperature Rise to Cook-Off.

Correlation and Extrapolation

To make the extrapolation for a 500-day cook-off temperature for any size of interest, the following is a correlation and extrapolation procedure that is used at NWC:

1. The experimental cook-off data are plotted as the logarithm of the equivalent time-to-reaction versus the reciprocal of the absolute temperature of the oven. Straight lines are drawn through the points for each size considered. This usually is a family of parallel lines (Figure 12).

2. A point is located on each line that is the oven temperature, T_1, at which $t_e = \tau$, a "thermal time constant" defined as

$$\tau = \lambda^2/\alpha \tag{16}$$

3. A straight line drawn through these $t_e = \tau$ points is the line from which the extrapolation is to be made.

4. At the $t_e = \tau$ point for the size of explosive charge being considered, a line is drawn parallel to the experimental slow cook-off lines and extended to the 500 day, 4.32×10^7 seconds, time to reaction.

5. The temperature, as $1/T_1$, at t_e = 500 days, is the desired value.

An "experimental" critical temperature is derived for each size considered using the empirical function of Zinn and Rogers[11] (Appendix A):

$$t_e/\tau = f(E^*/T_{cr} - E^*/T_1) \tag{17}$$

The function was derived from the cook-off behavior of a series of different explosives. (The function is represented in Figure 3 of reference 11). Figure 1 in Appendix A of this report is a reproduction of that figure.) At $t_e = \tau$, the function has a value of 1.6 for infinite cylinders, $L/D > 2$, and 1.0 for equi-cylinders, $L/D \cong 1$, so that

$$E^*\left(\frac{1}{T_{cr}} - \frac{1}{T_1}\right) = 1.6 \qquad (L/D > 2) \tag{18}$$

[11] T. Zinn and R. N. Rogers, *J. Phys. Chem*, Vol. 66 (1962), p. 2646.

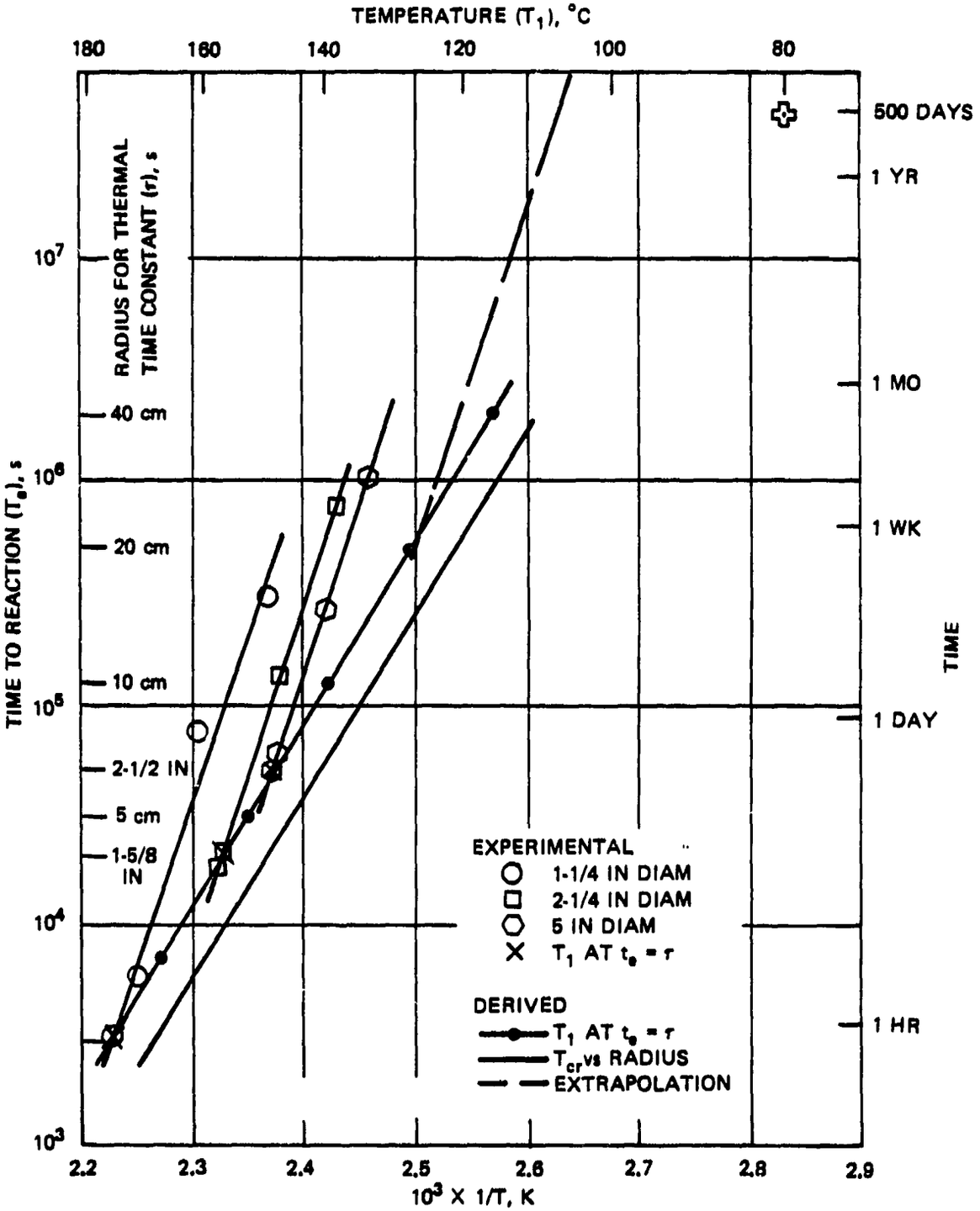

FIGURE 12. Slow Cook-Off Data Correlation and Extrapolation.

The $t_e = \tau$ line is described by the application of Equation 17 with an equation defining the characteristic "critical" temperature, T_{cr}, the λ/ρ form of Equation 9:

$$T_{cr} = \frac{E^*}{2.303\, R \log\left[\frac{a^2 \rho E^* A Q}{\lambda R T_{cr}^2 \delta}\right]} \tag{19}$$

whose major terms are the Arrhenius reaction rate parameters E^* and A. The slope of the $t_e = \tau$ line is primarily a function of the activation energy and the intercept is a function of the frequency factor. Usually, no more than a small adjustment is needed to fit the $t_e = \tau$ line.

FAST COOK-OFF–SMALL-SCALE COOK-OFF BOMB TEST

The SCB test provides a means for predicting the response of an ordnance item to fast cook-off, as in an aircraft fuel fire, using about 2 pounds of an explosive or propellant. The 2-pound sample size is adequate to provide the thermal gradient in the charge for the skin burn-off reaction to occur. This is characteristic of the reaction to a fire that occurs in the larger charges in shells, warheads, rocket motors, and bombs. In addition to determining time and temperature at which the runaway reaction occurs, the SCB, by heating the unit to destruction in each test, can be used to describe the reaction that occurs in a thermal runaway. The SCB also provides a means of assessing the effectiveness of proposed liner materials since the proposed liner can be applied to the SCB before loading.

The terms used to describe the reaction that occurs (detonation, explosion, or deflagration) are described in this section from the form of the fragments of the SCB unit. An official set of definitions given in MIL-STD-1648(AS) and described by reactions in Mk 82 GP bombs are given in Appendix B.

Experimental

The SCB unit (Figures 13 and 14) consists of a 400 milliliter stainless steel vessel with 3.2 millimeter thick walls. A 10-ohm length of nichrome ribbon is wound on an insulating layer of mica on the cylindrical surface, fastened with glass tape, and covered with 1 to 2 centimeters of lagging. The unit has a mild-steel cover with two feed-through fittings for the thermocouple leads and for a pressure take-off. The mounting frame consists of two 13.5 by 13.5 by 1.25-centimeter mild-steel plates with four 1.25-centimeter bolts that clamp the SCB unit between them. The unit is instrumented with one or more plate-type thermocouples; one is spot welded to the center of the vessel wall and another, if needed, is placed in the liner-explosive interface. The plate-type thermocouple consists of a 0.3-millimeter-thick nichrome

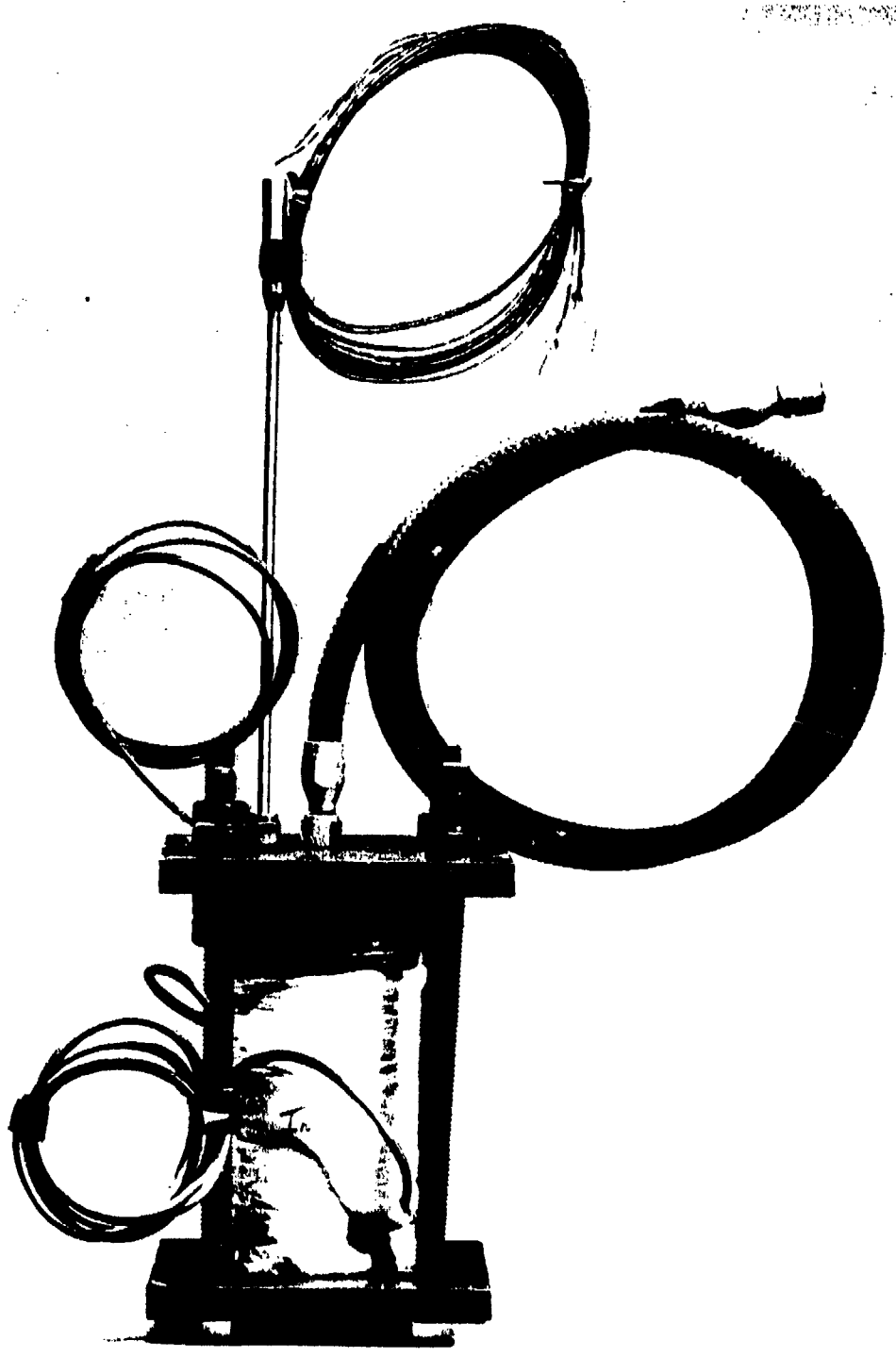

FIGURE 13. Small Scale Cook-Off Bomb (SCB) Assembled Unit. (Neg. LHL 182486)

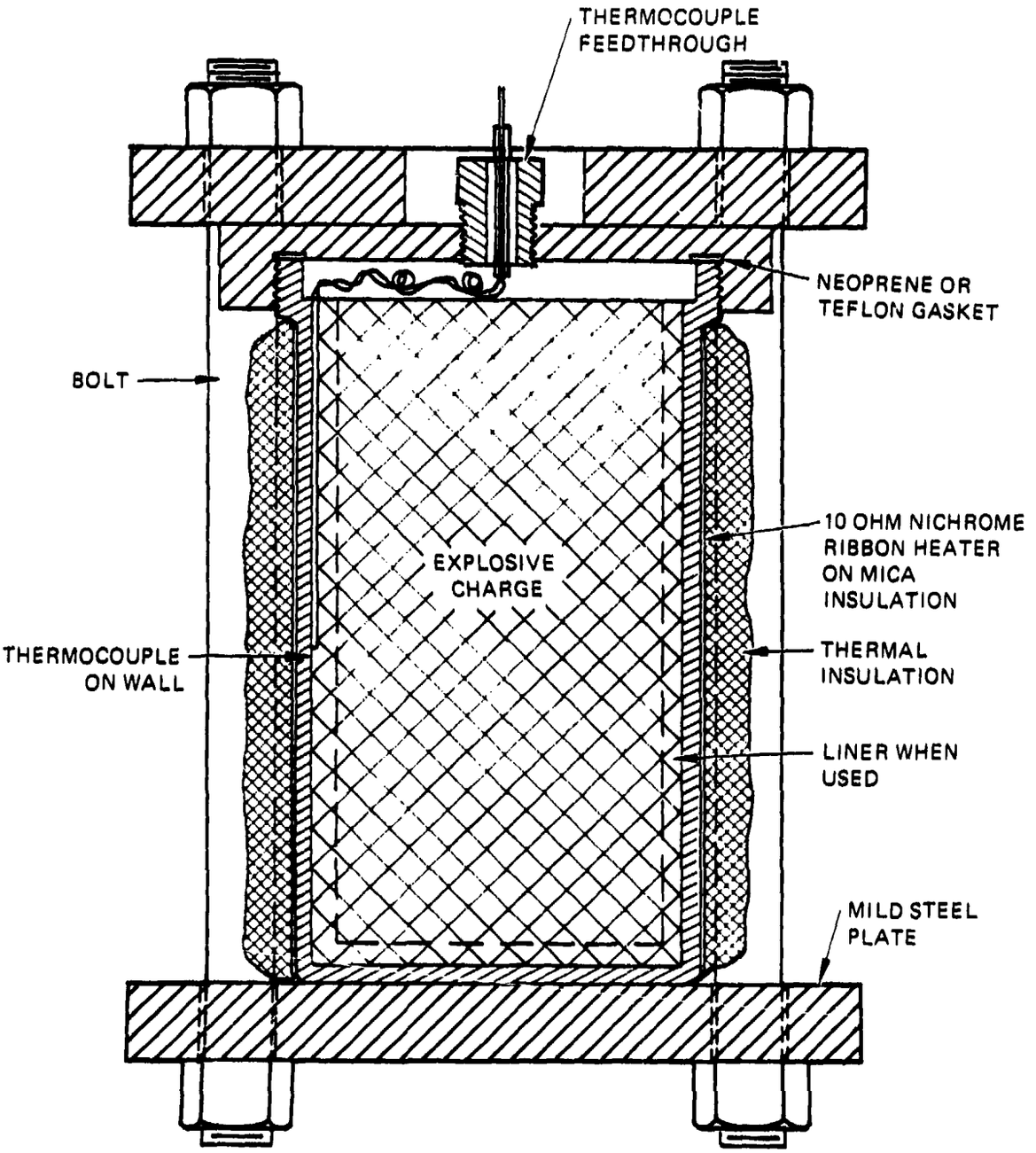

FIGURE 14. Cross-Section Sketch of SCB (Scale: Approximately Full Size).

ribbon approximately 1 centimeter square with the thermocouple wires fanned out and individually spot welded to the tab. If a controlled temperature is needed, another plate-type thermocouple can be placed under the mica insulation on the outside of the vessel to provide the control signal. Plate-type rather than bead-type thermocouples are used in the SCB since plate-type thermocouples give faster response and more representative measurement of the temperature at the interfaces.

The SCB unit is typically loaded to within 6 millimeters of the top by direct casting or with machined billets that fit snugly into the container. A small groove is cut into the side of a machined billet for passing the thermocouple leads. The unit is assembled as follows:

1. Thermocouple leads are attached to the feed-through wires.
2. Cap is screwed on.
3. Unit is assembled into the clamping frame.
4. Pressure hose (6,000 psi) is attached to the pressure feed-through. (The hose is approximately 2 meters long with a pressure transducer at the end.)
5. Assembled unit is installed in a bomb proof bay.
6. Power and indicator leads are connected.

Thermocouple, pressure transducer, and power leads are remotely checked for continuity. Fast strip-chart recorders are usually sufficient for this system. Immediately before time zero, the recorders are turned on; at time zero the power is turned on. Continuous readings are made of thermocouple and pressure transducer outputs until the SCB is destroyed by the fast cook-off reaction. The 115 VAC lines produce a heating rate of 2 to 3°C/s, the rate obtained in a bomb or warhead in a fire. Slower heating rates can be obtained by lowering the input voltage or by using a programmable recorder-controller. After the reaction, the fragments of the unit and the base plate of the clamp are recovered and examined.

Data Reduction

Time and temperature of the explosive reaction are taken from the chart records and an assessment of the severity of the reaction is made from the number and condition of the SCB fragments and the condition of the base plate. Levels of reaction severity usually identified are:

1. **Deflagration.** The SCB is in one or two pieces and the base plate is undisturbed (Figure 15).

2. **Explosion.** The SCB has broken up into a few or into many, often heat discolored, badly deformed pieces depending upon the violence of the explosion. The base plate is undisturbed or but slightly bent (0.0 to 0.5 millimeter) (Figures 16 and 17).

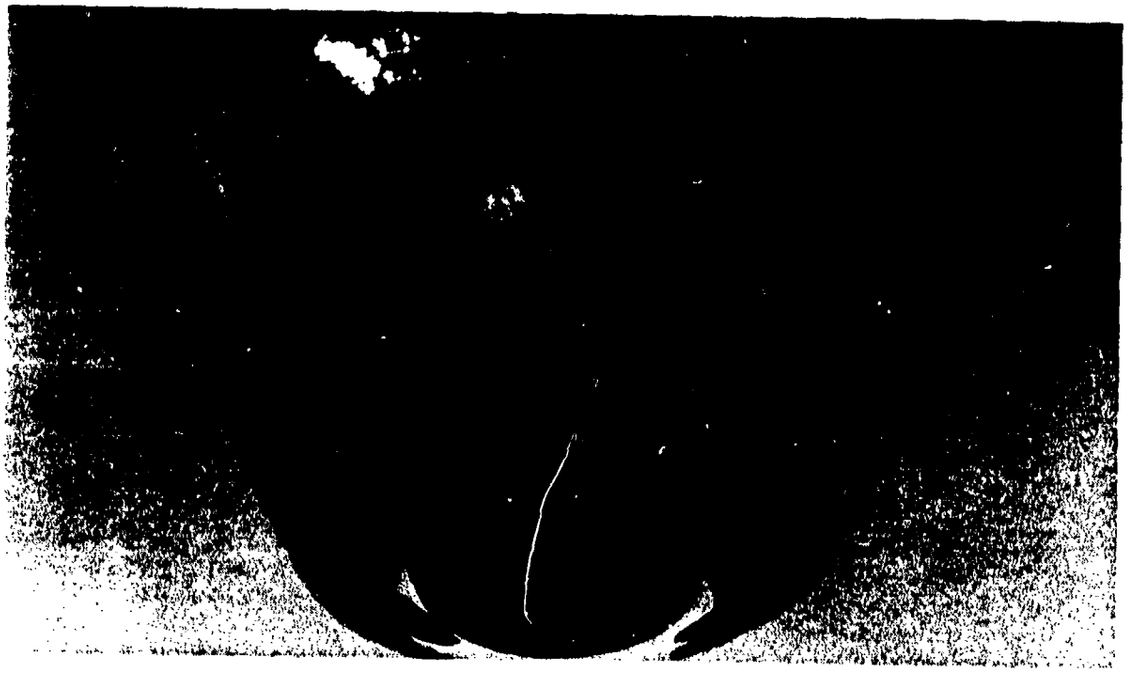

FIGURE 15. SCB Test Result, Deflagration. (Neg. LHL 184533)

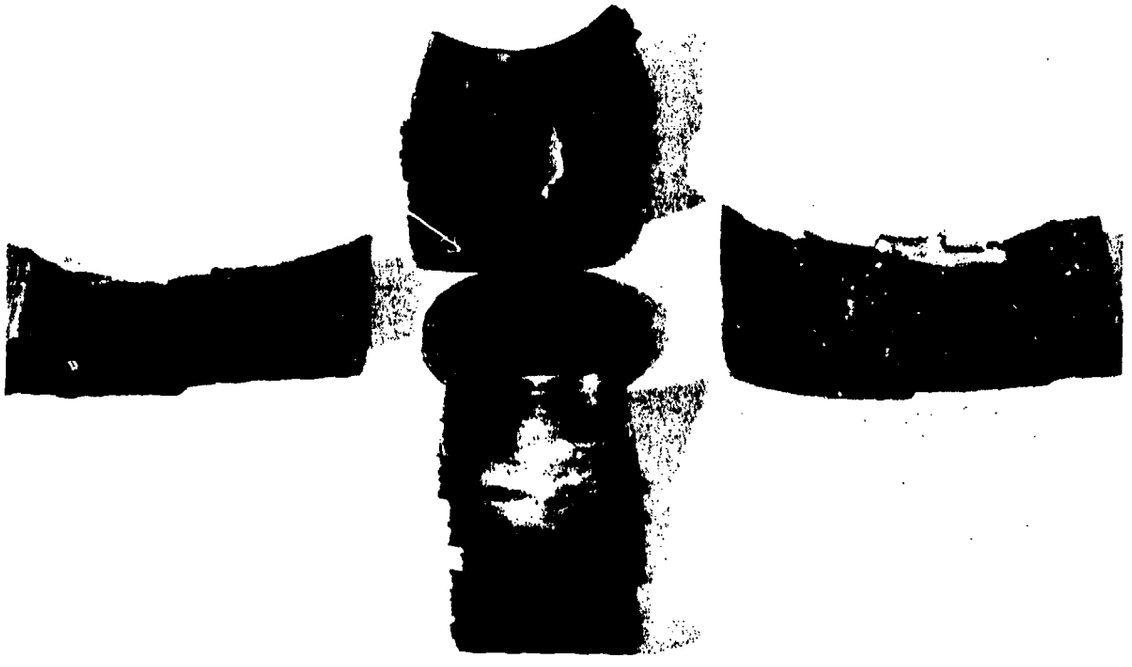

FIGURE 16. SCB Test Result, Mild Explosion. (Neg. LHL 184531)

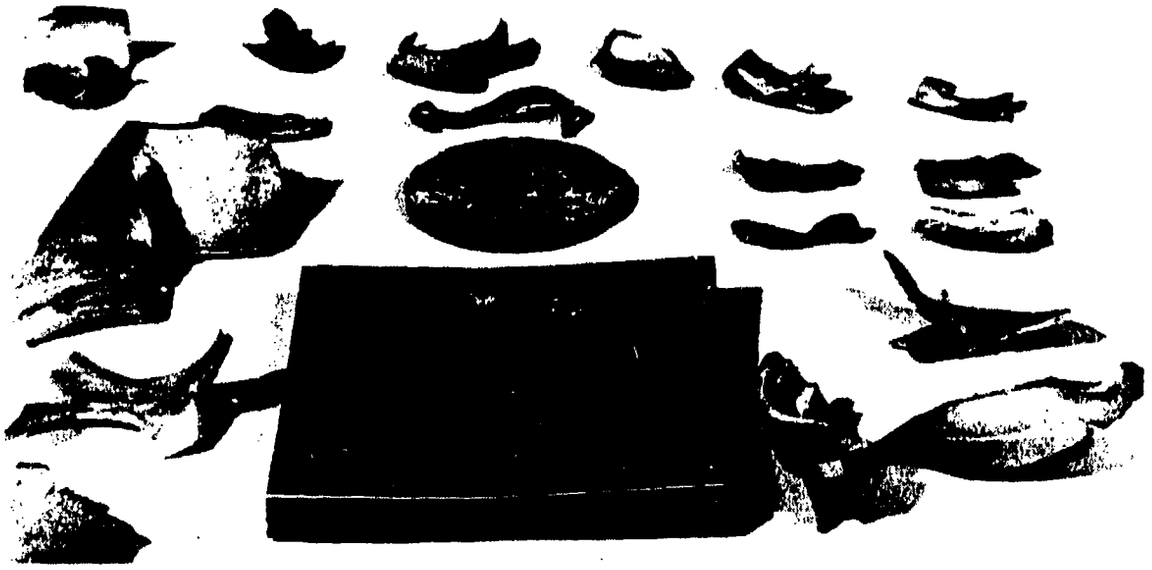

FIGURE 17. SCB Test Result, Violent Explosion. (Neg. LHL 190443)

3. **Detonation.** The SCB has broken up into many heat discolored fragments and the base plate is dented, if not punched through (Figure 18). An observer in a safe location would hear the reaction as a sound ranging from the muffled bang of a deflagration to the sharp crack of a detonation.

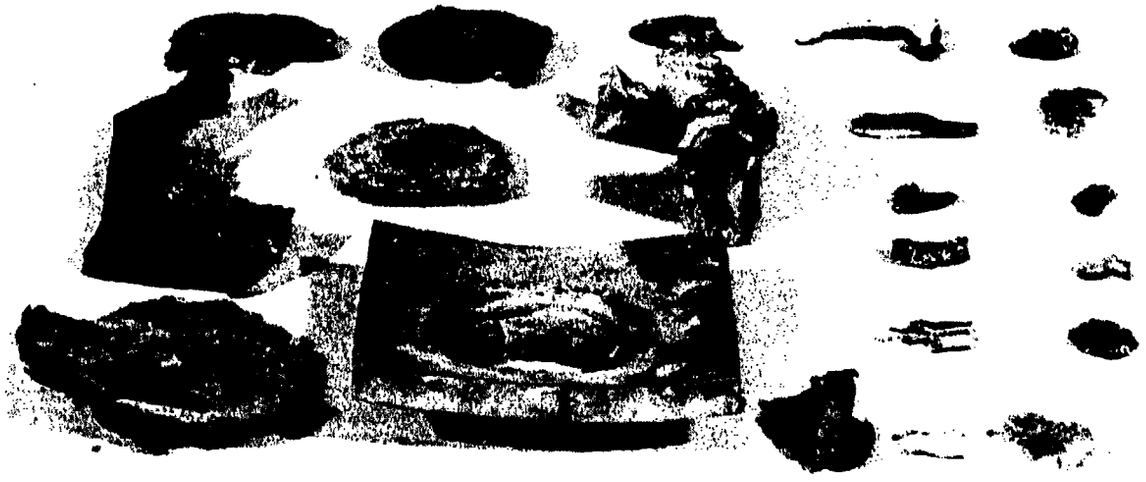

FIGURE 18. SCB Test Result, Detonation. (Neg. LHL 189103)

Criterion for Acceptance

An explosive or propellant can be said to have passed this test if the reaction produced is no greater than the mildest explosion, i.e., the SCB unit is in three to five pieces. Greater confinement of many munitions will certainly increase the severity of the reaction on fast cook-off.

FAST COOK-OFF--FULL-SCALE

The principal full-scale test of concern here is the fast cook-off in an aircraft fuel fire. Naval Weapons Requirement WR-50 outlines a series of tests for the final qualification of munitions; MIL-STD-1648(AS) was added to assure the safety of the munition in an accidental fire. Appendix B lists definitions of terms used to describe cook-off reactions.

A preliminary fuel fire cook-off experiment that uses but 12 to 15 pounds of explosives is available. This experiment has the advantage of using some but not all of the scale-up batch that is required for interim qualification of the explosive. The vehicle used is the 5-inch Mk 24 Zuni warhead. The 0.4-inch walls of this warhead provide sufficient confinement to obtain a response similar to that of larger weapons in a fuel-fire cook-off. The SCB and the Mk 24 Zuni warhead tests supply enough data so that the final, and costly, full-scale test can be limited to one or two confirmatory tests.

In this test, the loaded 5-inch Mk 24 Zuni warhead is suspended from an A-frame over a 12 foot octagonal fire pit, such that the warhead is 30 to 36 inches above the fuel. The warhead is suspended on chains attached to lift rings fitted into the fuse cavities at the nose and tail. Sufficient aircraft fuel is used to provide a fire for 10 minutes or more. A few gallons of gasoline floated on the surface of the fuel and electric matches wrapped with gasoline-soaked rags are used to initiate the fire. Thermocouples spaced around the warhead monitor the fire. Motion picture cameras operating at 400 and 24 feet per second record the fire and any reaction that occurs. Closed circuit television is used to visually monitor the test.

Observations via the visual monitors, the motion picture film, the fire time and temperature record, and still photographs taken after the test represent the test data. The violence of the reaction that occurred is the primary consideration of this test. Figure 19 shows a desirable result obtained with an experimental explosive in which the Mk 24 warhead was simply split open and is still on the A-frame.

A full-scale test of a main charge explosive would be made in a vehicle such as the Mk 81 or Mk 82 GP bomb containing about 100 or 200 pounds of explosive, respectively. The test arrangements are identical as in the smaller Mk 24 Zuni warhead test, except that a larger fire in a 24-foot octagonal pit is used for the larger items. (Appendix B shows the range of results of reactions in Mk 82 bombs with various explosives

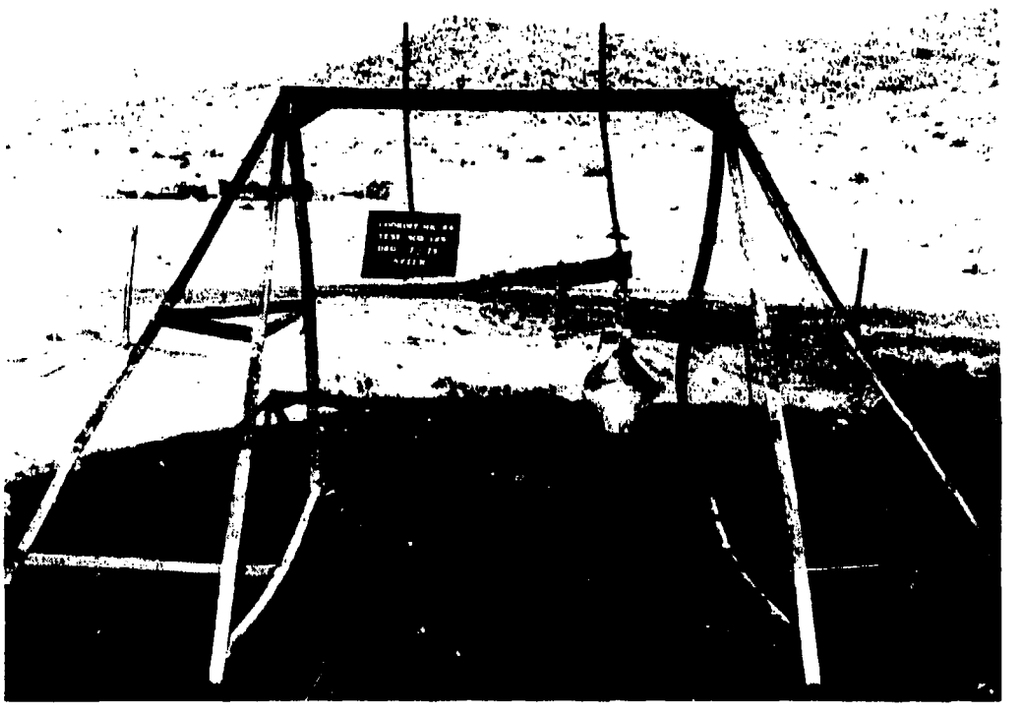

FIGURE 19. Full Scale Fast Cook-Off Test Result. Deflagration of Mk 24, Zuni warhead; experimental explosive. (Neg. LHL 179887)

and liners). Figure 20 shows results of a fuel fire fast cook-off in a Mk 81 GP bomb loaded with the same experimental explosive as was the Mk 24 Zuni warhead shown in Figure 19.

FIGURE 20. Full Scale Fask Cook-Off Test Result. Delfagration of Mk 81 G.P. bomb; experimental explosive. (Neg. LHL 182248)

Appendix A
SOURCES OF EQUATIONS USED FOR SLOW COOK-OFF

Many materials, including propellant and explosives, exhibit the phenomenon of self-heating. This is the liberation of energy in the interior of the material caused by a chemical reaction that is often a decomposition reaction. If this heat is not removed from the material, the temperature of the material increases with a concurrent increase in the rate of reaction. Eventually a dynamic equilibrium will be reached in which heat is removed as fast as it is generated; or, if the heat cannot be removed fast enough, the temperature and reaction rate increase until the material catches fire or explodes, if it is capable of doing so.

In the typical slow cook-off test, the time-temperature record (Figure A-1) shows three regions of interest for data reduction and for the predictions to be made.

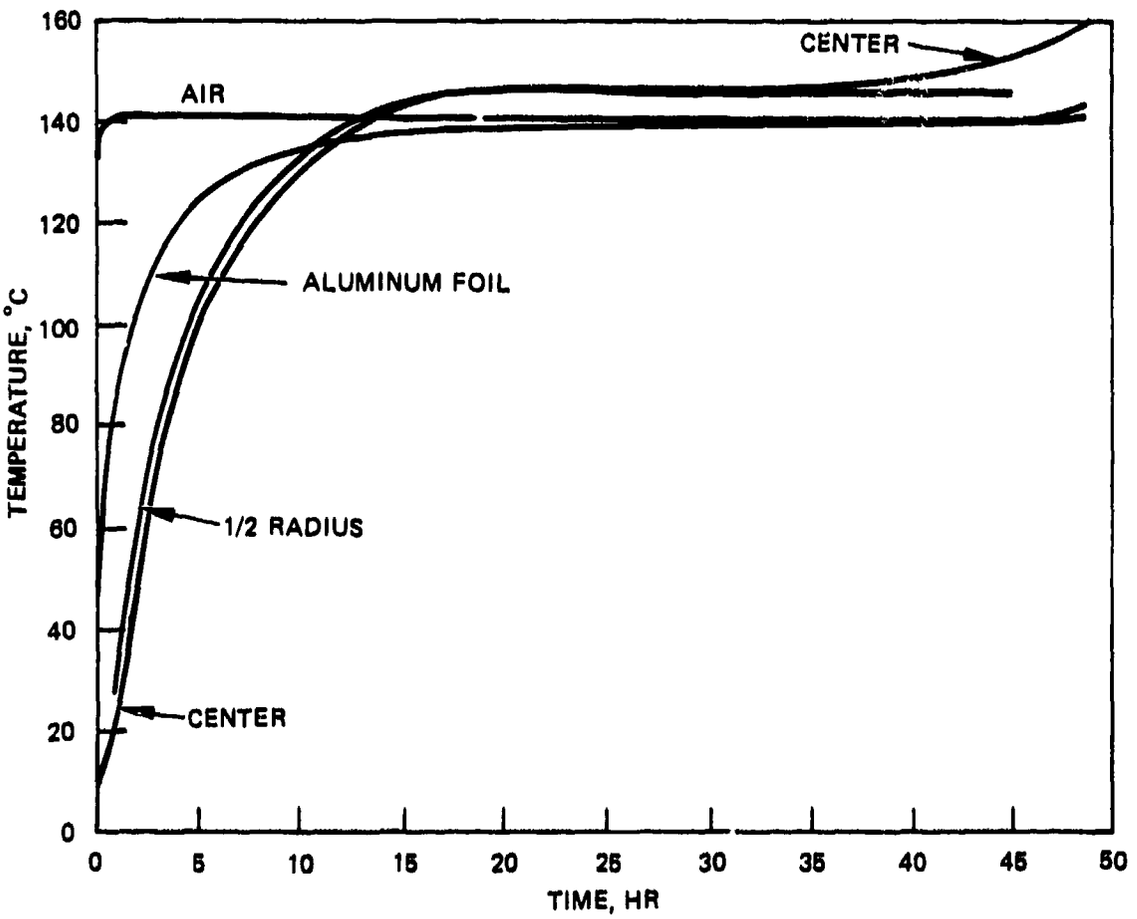

FIGURE A-1. Slow Cook-Off Time-Temperature Data. Experimental explosive 5-inch diameter by 5-1/2 inches long.

During the warm-up, as the sample comes up to the oven temperature, the temperature difference between the surface and center-of-sample thermocouples provides thermal diffusivity data. The second region is the quasi steady state where heat production balances heat loss to the oven. Finally, a third region is identified with an essentially adiabatic temperature rise to cook-off. These regions can be identified with solutions to the general heat flow equation with an internal heat source:

$$\rho c \frac{\partial T}{\partial t} = \lambda \nabla^2 T + \rho Q \frac{dx}{dt} \tag{A-1}$$

where dx/dt is a chemical reaction rate that is described by

$$\frac{dx}{dt} = k(1 - x)^n = (1 - x)^n A \exp(-E^*/RT) \tag{A-2}$$

Combining Equation A-2 with Equation A-1 gives the heat-flow equation to be solved:

$$\rho c \frac{dT}{dt} = \lambda \nabla^2 T + \rho Q A \exp(-E^*/RT) = \lambda \nabla^2 T + \rho Q k \tag{A-3}$$

During the warm-up period the material is essentially inert so that $\rho Q \approx 0$. In the steady state, $dT/dt = 0$, and during the adiabatic rise to cook-off, $\lambda \nabla^2 T \cong 0$.

During the warm-up period, the reaction rate at the low temperature is small so that little or no energy is produced and the ρQ term approaches zero:

$$\rho c \frac{dT}{dt} = \lambda \nabla^2 T \tag{A-4}$$

In a cylinder, $\nabla^2 T$ becomes unidimensional[12] and Equation A-4 integrates to

$$\Delta T = \frac{a^2}{4\alpha} \frac{dT}{dt} \tag{A-5}$$

Thus, thermal diffusivity, α, can be derived from the temperature difference, ΔT, during warm-up, leading to thermal conductivity by the definition

$$\lambda = \rho c \alpha \tag{A-6}$$

Analytic solutions of Equation A-3 for the steady state in a slab, cylinder, and sphere, have been obtained.[10,13-15] Following Longwell's derivation (footnote 10)

[12] I. B. Cadoff and R. Miller. *Thermoelectric Materials and Devices.* New York, Reinhold Publishing Corp., 1960, p. 131.
[13] D. A. Frank-Kamenetski. *Acta Physicochim (USSR)*, Vol. 10 (1939), p. 365.
[14] P. L. Chambre. *J. Chem. Phys.*, Vol. 20 (1952), p. 1795.
[15] P. H. Thomas. *Trans-Faraday Soc.*, Vol. 54 (1958), p. 10.

using the steady-state condition of $dT/dt = 0$ and an infinite heat transfer coefficient, Equation A-3 becomes

$$Qk_0 \cong \frac{4\lambda}{\rho a^2}(T_c - T_1) \tag{A-7}$$

for small arguments, i.e., zero order reaction or small fraction reacted. Frank-Kamenetski's solution of Equation A-3 (footnote 12) derives a "critical temperature," T_{cr}, for heat balance between heat generation in the sample and the loss to the surroundings.

$$T_{cr} = \frac{E^*}{2.303\ R \log\left[\dfrac{\rho a^2 QAE^*}{\lambda R T_{cr}^2\ \delta}\right]} \tag{A-8}$$

This characteristic critical temperature is a useful criterion to describe a thermal property of a particular explosive in a given application.

Longwell (footnote 10) treated the third case of the essentially adiabatic temperature rise to cook-off. The adiabatic condition obtains during the rapid rise due to the low thermal conductivity of these materials. With the term $\lambda\nabla^2 T = 0$, Equation A-3 becomes

$$\rho c \frac{dT}{dt} = \rho Q k_0 = \rho QA \exp(-E^*/RT) \tag{A-9}$$

providing another approach to chemical reaction rate information from experimental data.

A further, and most useful, development from the steady state approach is the work of Zinn and Rogers (footnote 11), who found an empirical relation between time-to-cook-off and temperature that described the cook-off behavior of a number of different explosives. They found that the ratio of time to cook-off, t_e, to a "thermal time constant," τ, defined as a^2/α, is a function of $E^*(1/T_m - 1/T_1)$.

$$t_e/\tau = f\left(\frac{E^*}{T_m} - \frac{E^*}{T_1}\right) \tag{A-10}$$

Values of the function are presented as a plot versus the log t_e/τ in Figure 3 of reference 11 and is reproduced here has Figure A-2. Equations A-8 and A-10 provide equations for the extrapolations required for the qualification of explosives.

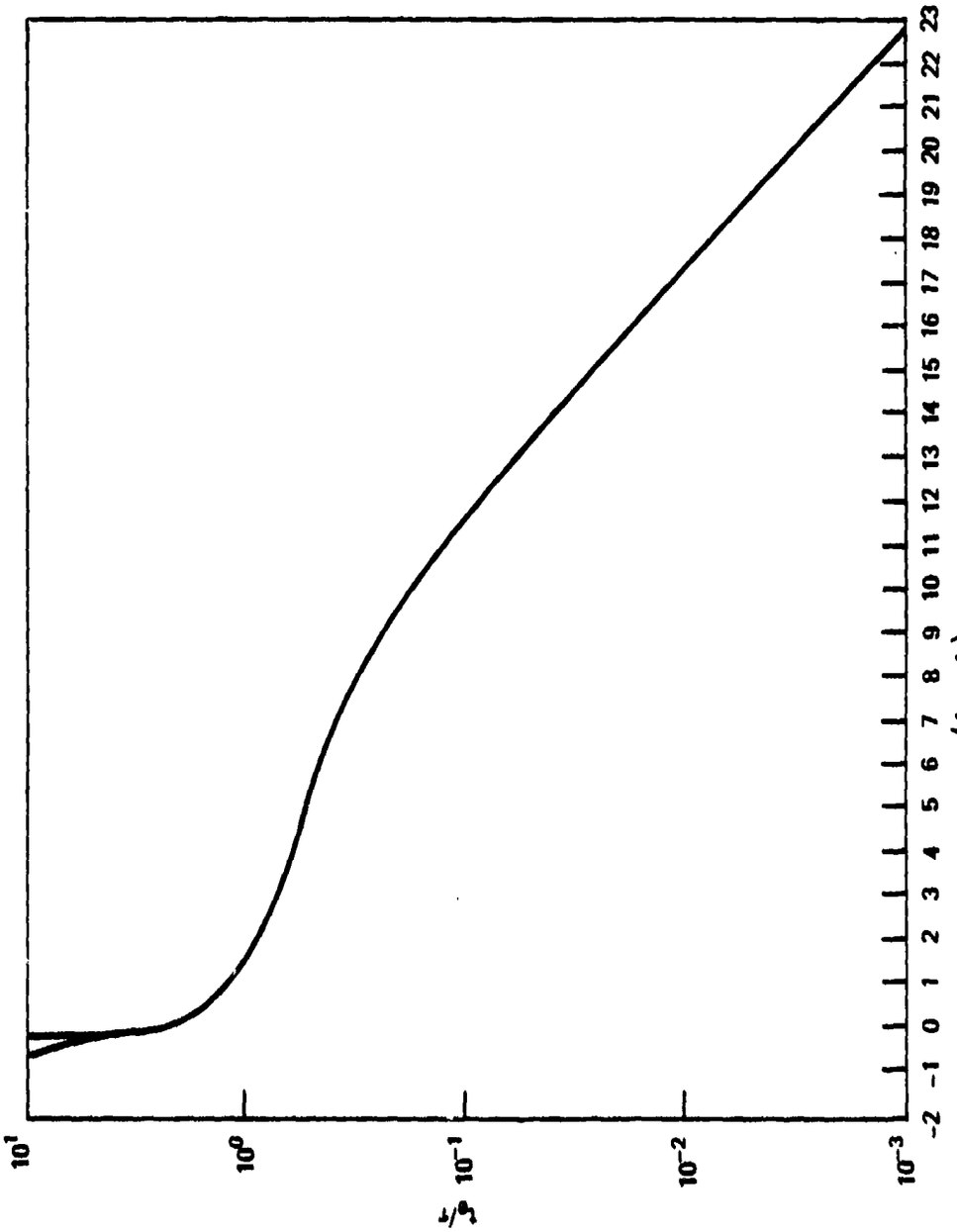

FIGURE A-2. Plot of Equation A-9.

Appendix B
DEFINITIONS OF ORDNANCE EXPLOSIVE REACTIONS OBTAINED ON COOK-OFF

The Naval Ordnance Systems Command and the Naval Air Systems Command established the following terminology and definitions to be used when reporting ordnance explosive reactions resulting from cook-off. They are listed in decreasing order, based on air shock and fragmentation. Items 1, 2, and 3 are reprinted from NAVORDNOTE 8020, 23 April 1969; items 4 and 5 are reprinted from NASC MIL-STD-1648(AS).

1. **Detonation.** Munition performs in design mode. Maximum possible air shock formed. Essentially all of case broken into small fragments. Blast and fragment damage is at maximum. Severity of blast causes maximum ground crater or flight deck hole capable by the munition involved.

2. **Partial Detonation.** Only part of total explosive load in munition detonates. Strong air shock and small as well as large case fragments produced. Small fragments are similar to those in normal munition detonation. Extensive blast and fragmentation damage to environment. Amount of damage and extent of breakup of case into small fragments increase with increasing amount of explosive that detonated. Severity of blast could cause large ground crater, or large flight deck hole on carrier if munition is large bomb; hole size depends on amount of explosive that detonates.

3. **Explosion.** Violent pressure rupture and fragmentation of munition case with resulting air shock. Most of metal case breaks into large pieces which are thrown about with unreacted or burning explosive. Some blast and fragmentation damage to environment. Fire and smoke damage as in deflagration. Severity of blast could cause minor ground crater, or small depression of flight deck or carrier if munition is large bomb.

4. **Deflagration.** The process where the ordnance energetic material undergoes rapid combustion and ruptures its enclosure. The item or major parts thereof may be thrown up to 50 feet by the reaction. No damage due to blast effects or fragmentation. Fire fighting capability may be encumbered by expansion of fire and burning material and parts being thrown about.

5. **Burning.** The process where the ordnance energetic material undergoes combustion. During this reaction, the ordnance case may open up and vent. The item remains in position although it may fall due to structural failure. The burning reaction presents no hazard to fire fighting capability.

Figures B-1 through B-5 show results of Mk 82 bomb cook-off reaction that illustrate the definitions listed above.

FIGURE B-1. Summary View of Mk 82 Bomb Cook-Off Test Results.
(Neg. LHL 180243)

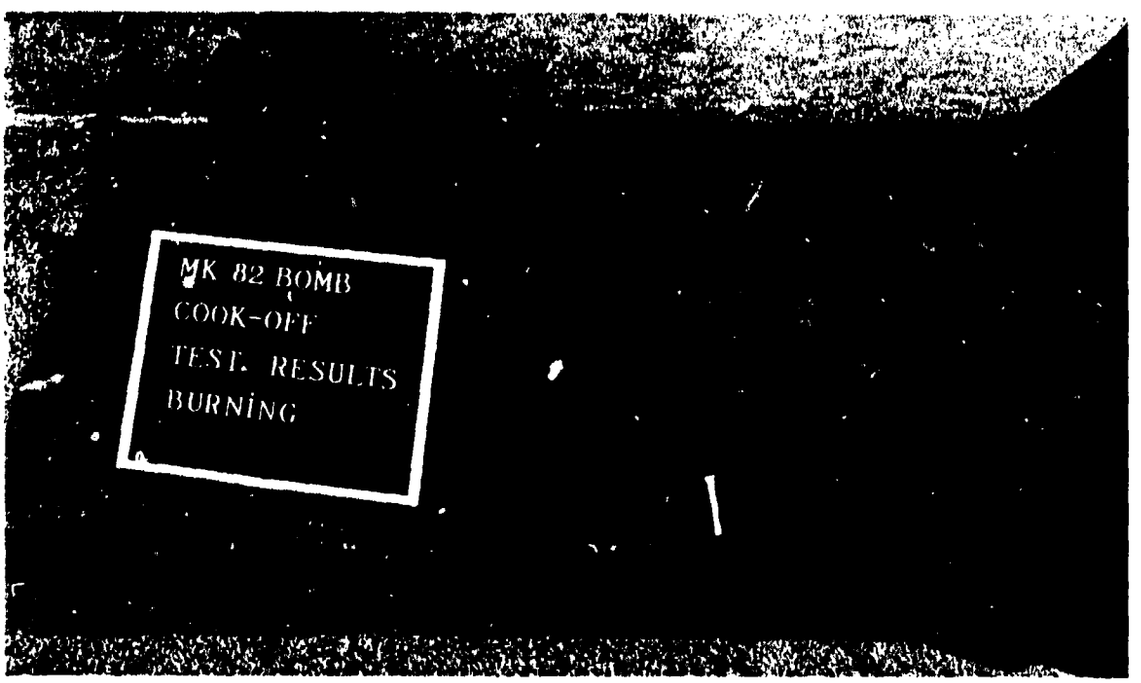

FIGURE B-2. Mk 82 Bomb Cook-Off Test Result: Burn.
(Neg. LHL 180240)

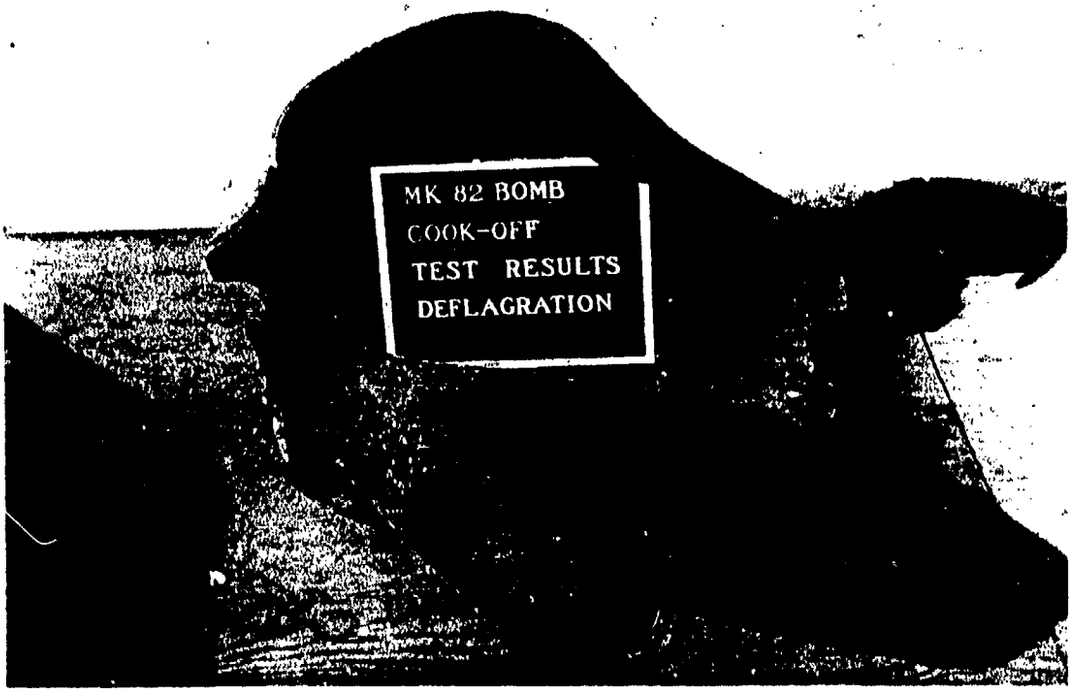

FIGURE B-3. Mk 82 Bomb Cook-Off Test Result: Deflagration. (Neg. LHL 180241)

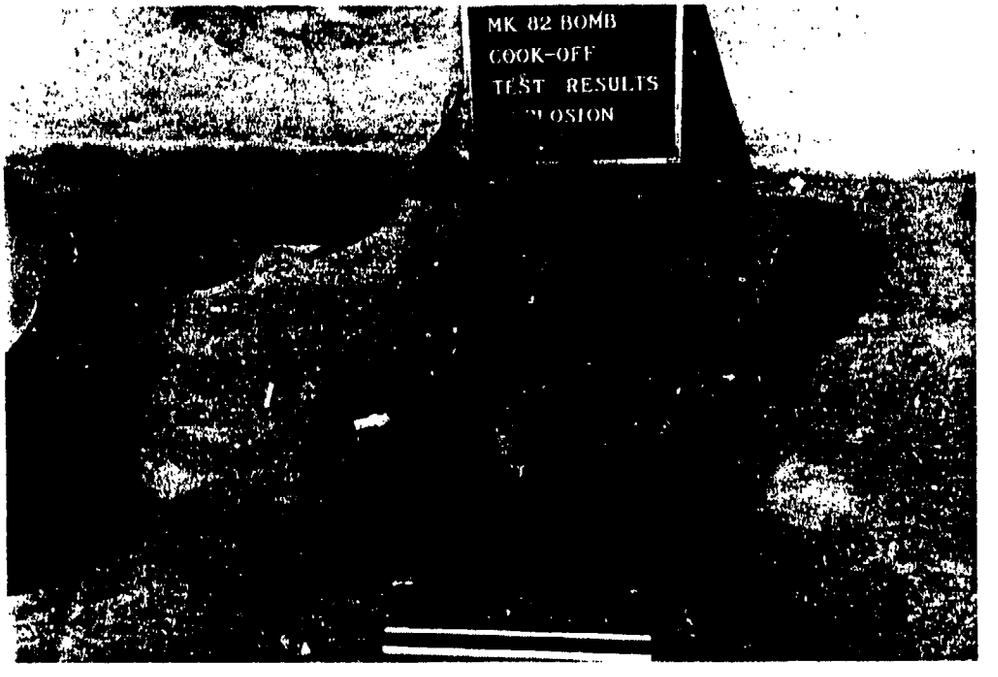

FIGURE B-4. Mk 82 Bomb Cook-Off Test Result: Explosion. (Neg. LHL 180239)

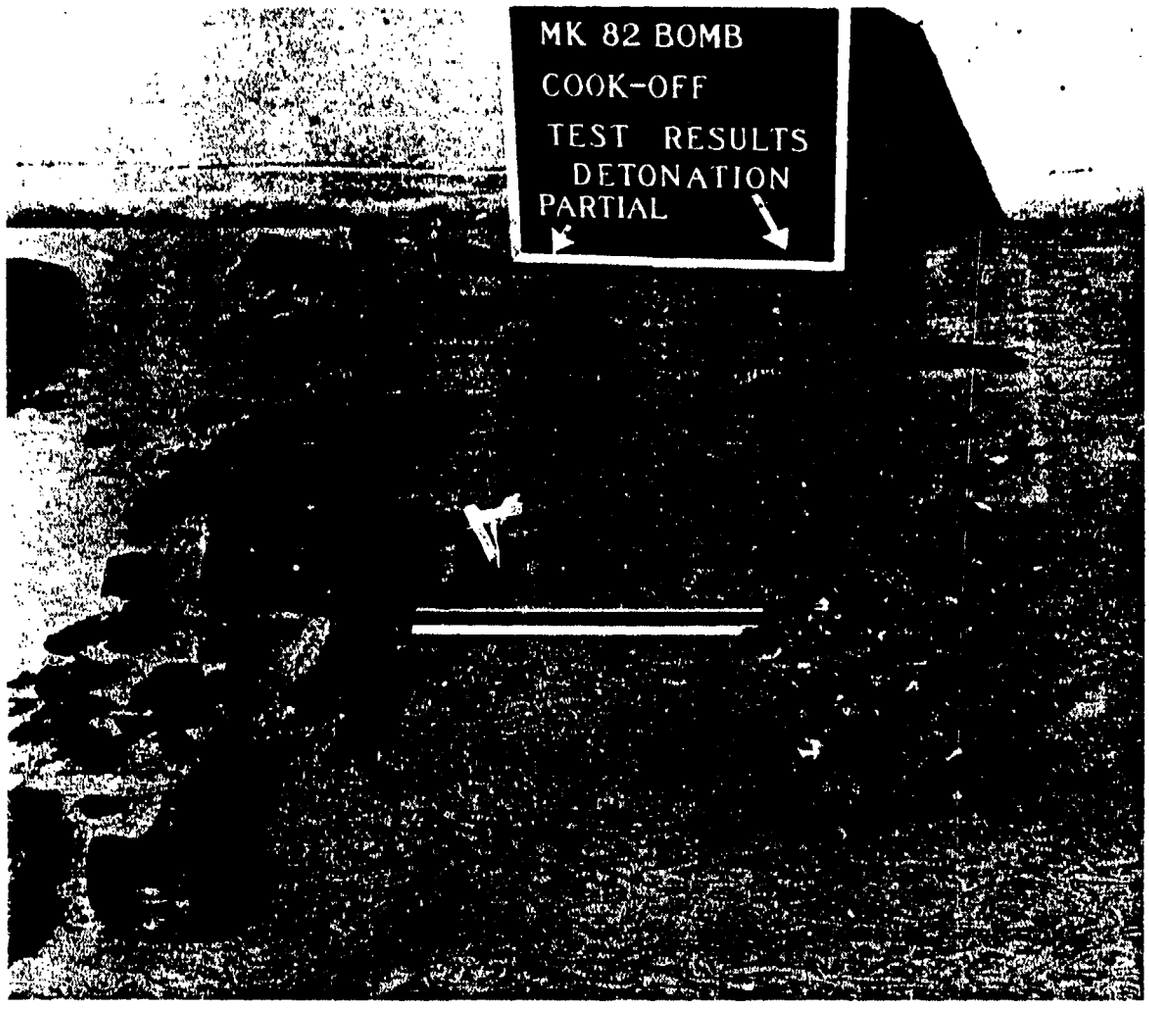

FIGURE B-5. Mk 82 Bomb Cook-Off Test Result: Partial Detonation and Design Mode Detonation. (Neg. LHL 180242)

NOMENCLATURE

λ	Thermal conductivity, cal/s-cm•°C
∇	Differential operator
T	Temperature, K
ρ	Density, g/cm^3
c	Specific heat, cal/g•K
k	Specific rate constant, s^{-1}
t	Time, seconds
Q	Heat of reaction, cal/g
A	Arrhenius frequency or pre-exponential factor, s^{-1}
E^*	Activation energy, cal/mol
R	Gas constant, cal/mol•K
T_c	Temperature at sample center, °C
T_{cr}	Critical temperature, °C
T_m	Temperature of maximum reaction, °C
a	Characteristic dimension, radius or half thickness, cm
δ	Shape factor, 0.87 for slabs, 2 for cylinders, 3.32 for spheres
t_e	Time to reaction, seconds
τ	Thermal time constant, = a^2/α, seconds
α	Thermal diffusivity, = $\lambda/\rho c$ cm^2/s
x	Fraction of material reacted
n	Reaction order
T_1	Temperature of surroundings (oven), °C
w	Weight of sample
w_0	Initial weight of sample

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